

# CHARACTERISATION AND BENEFICIATION OF HIGH-ALUMINA INDIAN IRON ORE FINES BY SELECTIVE FLOCCULATION

A Thesis Submitted  
In Partial Fulfilment of the Requirements  
for the Degree of  
MASTER OF TECHNOLOGY

By  
BELEHALLI GURURAJ

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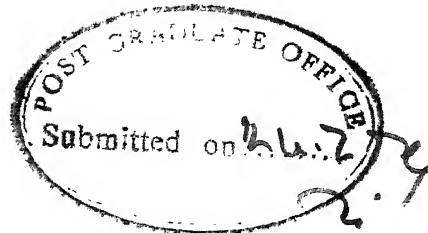
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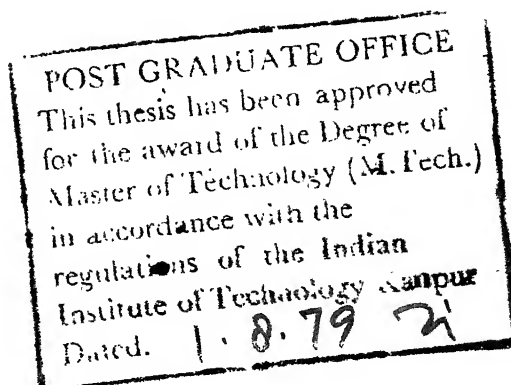
**CERTIFICATE**

Certified that the present work entitled, 'Characterisation and Beneficiation of High Alumina Indian Iron Ore Fines by Selective Flocculation' by Belhehalli Gururaj has been carried out under my supervision and has not been submitted elsewhere for any degree.

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(AUTHOR)



## ABSTRACT

Indian Iron ores have a peculiar problem of high Alumina content. Attempts have been made to define, analyse and solve the problem by different techniques.

At first some iron ores were characterised by x-ray analysis and by electron micrographs and diffractograms. Aluminium was found in the form of clay materials like, kaolinite, Illite and montmorillonite dispersed as fine grains.

Some flocculants were tried to achieve selectivity between aluminium-containing minerals and iron ore particles. Magnafloc series and Morarfloc series flocculants and Starch were employed. Both anionic and cationic type of flocculants were used. Single stage as well as multiple stage flocculations were tried.

Stability studies were carried out with a settling column. Settling characteristics of minerals were found out. Attempts were made to find out effect of dispersants on settling behaviour by using the same technique. The concentration dependence of dispersants on settling behaviour of minerals was found out.

Preliminary results show feasibility of selective flocculation technique for beneficiation of high-alumina Indian iron ore. More detailed studies are in progress.

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## CHAPTER I

### INTRODUCTION

The total inferred reserves of iron ores in India exceed 12,000 million tonnes assaying on the average over 52 pct iron<sup>1</sup>. A substantial amount of this reserve is hematitic and high-grade in terms of iron content. However, the problem with Indian iron ores is two-fold. One is the high content of gangue materials particularly aluminium-containing minerals, the other being the relatively soft nature of the ore which generates fines on handling. Alumina is believed to exist in Indian iron ore especially as lateritic clay, formed by the decomposition of basic rocks under tropical conditions.

Indian iron ore contains 1-7 pct.  $Al_2O_3$  which increases the viscosity of the slag in the Blast furnace. To counteract this, lot of dolomite stone and quartzite have to be added. This does in turn result in an increase in coke rate, contamination level and silicon in hot metal. The ore deposits in India normally have  $Al_2O_3/Fe$  ratio around 0.05-0.08 or more. It has been estimated that for a drop in the  $Al_2O_3/Fe$  ratio by 0.01 in the beneficiated ore, the cost per ton of hot metal produced is reduced approximately by Rs. 7.50 through increased production of hot metal in the blast furnace and reduced coke consumption. For efficient blast furnace operation, it is

also necessary to keep the  $\text{Al}_2\text{O}_3/\text{SiO}_2$  ratio to a low value.

Indian iron ore is of relatively soft nature, and the introduction of mechanization and heavy blasting of the mines is generating larger quantities of fines<sup>3-6</sup>. The proportion of -10 mm fines from Indian mines is never less than 35 pct. and sometimes reaches 50-60 per cent<sup>6</sup>. Blast furnace technology needs close sizing of the lump ores. Classification, preferably wet, generates substantial quantity of fines, which contain significantly high quantity of iron apart from increased percentages of silica and alumina. There are also large deposits of blue dusts or the natural fines in the lower layers of the deposits. Non-utilisation of the large quantities of fines would amount to national wastage. On the other hand, such fines should be beneficiated before pelletisation or sintering to produce agglomerates which are suitable as blast furnace burden material.

Attempts to wash Indian iron ores for the purpose of removal of alumina have met with only limited success<sup>3,4,7,8</sup>. In the Noamundi Washing plant<sup>3,8</sup>, the feed containing 58.8 pct. Fe, 3.54 pct.  $\text{SiO}_2$  and 6.50 pct.  $\text{Al}_2\text{O}_3$  could be ground, washed, rinsed or deslimed to produce a concentrate assaying 59.5 pct. Fe, 3.10 pct  $\text{SiO}_2$  and 5.70 pct  $\text{Al}_2\text{O}_3$ . Thus,  $\text{Al}_2\text{O}_3/\text{Fe}$  ratio could be slightly reduced from 0.11 to 0.095 whereas  $\text{Al}_2\text{O}_3/\text{SiO}_2$  ratio

was actually increased from 1.66 to 1.84. The work on Rajharapahar ore gave similar results<sup>7</sup>. Quartz, the predominant gangue material could be easily removed by washing but not the ferruginous clay material containing aluminium. The ore assaying 54.8 pct Fe, 11.16 pct  $\text{SiO}_2$  and 4.46 pct  $\text{Al}_2\text{O}_3$  was washed and screened producing high-alumina fines and a concentrate assaying 60.73 pct Fe, 5.7 pct  $\text{SiO}_2$  and 2.57 pct  $\text{Al}_2\text{O}_3$ . This beneficiation step resulted in lowering of  $\text{Al}_2\text{O}_3/\text{Fe}$  ratio from 0.081 to 0.042 and yet increase in  $\text{Al}_2\text{O}_3/\text{SiO}_2$  ratio from 0.40 to 0.45.

The general Indian problem as stated above is a challenging one meriting deeper scientific investigation. It was felt that a basic solution could be found only if the mode of occurrence of aluminium in the ore was established. This information is not available at present. The specific minerals containing aluminium, their grain sizes, mesh of grind for liberation etc. should be determined, and some beneficiation technique applicable to fine particles-such as selective flocculation-should be adopted. This approach was undertaken in the present investigation and applied to the Barsua Iron mine samples, which represent a very typical Indian case.

Owned by the Rourkela Steel Plant (the geographical location of Rourkela in Orissa, India, is 22.25 N and 85.00 E), The Barsua iron ore deposit is located about 100 Km. from

Rourkela and supplies annually 2 million tonnes of iron ore to the steel plant. A part of the R.O.M. is fed to the washing plant (about 700 Tons per hour) which produces 34 pct + 10 mm. lump material, 37 pct -10+2 mm. fine suitable for agglomeration and more than 24 pct Slime<sup>2</sup>. A typical feed to the beneficiation plant assays 57.8 pct Fe, 2.8 pct SiO<sub>2</sub> and 7.2 pct Al<sub>2</sub>O<sub>3</sub>, the washed + 10 mm lump : 60.9 pct Fe, 1.9 pct SiO<sub>2</sub> and 4.9 pct Al<sub>2</sub>O<sub>3</sub> and the slime approximately assays 53.5 pct Fe, 6.5 pct SiO<sub>2</sub> and 9.2 pct Al<sub>2</sub>O<sub>3</sub>. Thus, the Al<sub>2</sub>O<sub>3</sub>/Fe ratio is brought down from 0.12 to 0.08 (which is not low enough) and Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> ratio does not shift appreciably from 2.6. The slime contains considerable quantity of iron oxide, separation of which from alumina is a challenging problem. Even dumping such a large quantity of material-a quarter of the feed to the washing and beneficiation plant-year after year imposes civil engineering and ecological problems<sup>2,6</sup>. Barsua iron ore thus presents a typical problem associated with fine-grained alumina-rich Indian iron ores.

There are many other high-alumina iron ores in India such as the one used in Bhilai, the one available in Byram, Andhra Pradesh etc.

Prior to selective flocculation studies, phase studies are most essential. Literature survey yielded no information regarding the mode of occurrence of aluminium in the hematitic iron ores of India. The complexity of the problem was

confirmed through the correspondences with the global experts on mineralogy (vide Appendix 'A'). It was therefore considered necessary that work should be done on phase characterisation of Barsua (Rourkela), Bhilai and other ores particularly of the Barsua deposit. The informations regarding size and phase composition of aluminium-rich grains would enable us to pursue the strategy of selective flocculation scientifically in obtaining high-iron and low-aluminium concentrates.

On the basis of the data obtained as above, selective flocculation experiments were designed with imported and indigenous polymeric flocculants of polyacrylamide type and also starch.

## CHAPTER II

### CHARACTERISATION OF BARSUA IRON ORE

Representative samples of Barsua Iron ore and the slimes generated in the washing and beneficiation plant were obtained through the courtesies of the Rourkela Steel Plant.

Iron, silica and alumina were analysed by conventional wet chemical methods. The other detected elements viz., Chlorine, Manganese and Potassium were estimated by  $\text{AgNO}_3$  method, permanganate colorimetric technique and flame photometry respectively.

Petrographic information regarding the samples was furnished by the SAIL<sup>2</sup>. X-ray diffraction studies on the powdered samples (- 200 mesh size) were carried out by the Debye-Scherrer technique and using a GEC X RD-6 diffractometer and employing Cr K  $\alpha$  radiation. On account of the unsatisfactory results from x-ray investigation, which were due to low proportions of fine grained aluminium - containing minerals, electron microscopic and microprobe studies were considered as essential and therefore undertaken.

The characterisation of very fine grains in the Barsua iron ore sample was carried out using the Cambridge Stereoscan and the JEM 120C electron microscope fitted with the ASID-4D (ultra-high resolution) scanning stage and Kevex Energy



Dispersive analyser. 10g. of the crushed ore were suspended in 100 ml acetone and subjected to ultrasonic vibration in an ultrasonic cleaner for 15 minutes. After allowing the suspension to settle, a drop of the supernatant liquid was introduced on a thin ( $\sim 500 \text{ \AA}$ ) carbon film supported by a 3 mm diameter 100 mesh size copper grid. The latter was mounted in the specimen stage (high resolution goniometer stage) of the JEM 120C electron microscope for studying the distribution of particles and identification by electron diffraction. For the micro-analytical investigation, a few drops of the supernatant liquid were deposited on a graphite support and the latter mounted in the stereoscan or in the ASID-4D stage.

Secondary emission images of the particles were obtained in the stereoscan at magnifications of upto X5000. At higher magnifications the images of the particles were poorly defined. The distribution of the elements in the particles was obtained by X-ray mapping of the regions containing particles using the wavelength dispersive crystal spectrometer attached to the Stereoscan (WDS). It was not possible to carry out chemical analysis using the WDS as the count rates for the elements Al, Si and K were very low ( $\sim 1$  to  $3/100$  sec.) Secondary emission images of the particles were obtained with the ASID-4D stage at accelerating voltages of 20 and 40 kV. Chemical analysis of the particles, using this stage, was carried out by tilting the specimen holder to between  $30$  and  $45^\circ$  with respect to the

electron beam and employing an electron probe ( $\sim 200 \text{ \AA}$ ), the finest one provided with the equipment. The characteristic x-rays emanating from the particles were characterised by the Kevex analyser and the results of intensity vs energy displayed on a TV screen or processed by a computer for arriving at the integrated intensities corresponding to various elemental peaks. A count time of 100 seconds was employed in all experiments. X-ray intensities from standards in the form of pure elements were used for atomic number, absorption and fluorescence corrections. About thirty regions and over 400 grains were scanned in order to characterise particles size distribution and elemental composition and to identify the phase constituting Aluminium-containing minerals in the sample.

Transmission electron microscopy was carried out at an accelerating voltage of 100 kV and micrographs of areas containing particles were recorded at magnifications X 10,000-X 20,000. The corresponding diffraction patterns were recorded at camera lengths of 76 and 120 cms. Sufficient care was taken to avoid photographing artefacts on the carbon film. The diffraction patterns obtained show the presence of both single and polycrystalline particles in the region covered by the diffraction aperture (an area of 0.23 micron diameter); occasionally fuzzy rings indicating the presence of amorphous phases were encountered. The values for interplanar spacings,  $d$ , were obtained from measurements of the radius  $R$  of the

rings in the case of polycrystalline samples or the distance  $R$  of the individual diffraction spots from the central spot for single crystal patterns and knowing the value for the camera constant,  $L\lambda$ , for the microscope obtained with the help of an evaporated gold film.

## RESULTS

A large number of R.O.M. analysis for Barsua provided the figures as 58-64% Fe, 1.5-2.0%  $\text{SiO}_2$ , 4-6%  $\text{Al}_2\text{O}_3$ . The 'slime' assayed 52-55% Fe, 5-7%  $\text{SiO}_2$  and 8-12%  $\text{Al}_2\text{O}_3$ . The composite R.O.M. sample also contained around 0.024% Chlorine, 0.66% potassium and 0.046% manganese.

Long exposure was given to obtain Powder diffraction photograph and identify trace minerals present (Fig. 1). Standard conversion table was used for getting the corresponding values of ' $d$ ' for each  $2\theta$  measured from the film. Hematite was found as the major element. Goethite in small amount and gibbsite and quartz in trace amounts were detected. This information given by the Geological Survey of India, Calcutta did not go beyond the petrographic information provided by the SAIL<sup>2</sup>, which did not mention any specific mineral containing aluminium.

Relative Intensity versus Diffraction Angle (starting with  $2\theta = 6^\circ$ ) curve or a diffractogram (Fig. 2) indicated presence of hematite, kaolinite, quartz and illite. Some

peaks were unidentified and a hump region indicated presence of amorphous clay minerals. Characteristic 'd' values for Fe and Al minerals obtained from specific references<sup>9-13</sup> are given in Appendix 'B'.

When the sample was subjected to citrate-bicarbonate-dithionite method of dissolution for hematite as suggested by Jackson<sup>14</sup>, (Vide Appendix 'C'), the residue showed heightened peaks for illite, kaolinite etc. The data alongwith these for standard mineral specimens are given in Fig. 3 and Appendix 'D'.

Electron micro-analytical studies and electron diffraction patterns provided conclusive information regarding the minerals containing aluminium. The elemental X-ray intensity peaks confirmed presence of not only Al and Si but also Mg, K, Cl etc. Typical regions shown in Figs. 4(a) and 4(b) give elemental composition to be 5:3:1:1 in terms of weight ratios for Si, Al, K and Mg respectively. It may be noted that composition of illite, though widely variable, may be typically 6.1:2.43:1.38:1 in terms of weight ratio of the above four elements. Figs. 4(c) and 4(d) show regions where Fe:Si:Al ratio were 7:1:1. Ignoring iron, Si:Al ratio was 1:1. In kaolinite, the corresponding ratio is very close to 1:1.

Few other micrographs (I, II and III) indicate the specific grains for which elemental abundance peaks were obtained: I-1 (Al, Si, K), 2 (Al, Si), 3 (very prominent Al); II-1 (Fe K $\alpha$  and K $\beta$ ), 2 (Al, Si, K) etc.

Another set of micrographs 604-612 (even numbers) were taken for Al-rich zones, corresponding to which electron diffraction patterns 603-611 (odd numbers) provided characteristic ring radii. 'd' values were computed for possible identification of phases. The measured and computed data are provided in Appendix E. The presence of kaolinite and illite were confirmed. Presence of quartz, montmorillonite, diaspora and gibbsite were also suspected. Table I and II summarise the information compiled.

TABLE ISize and Elemental Composition of Some Typical Grains

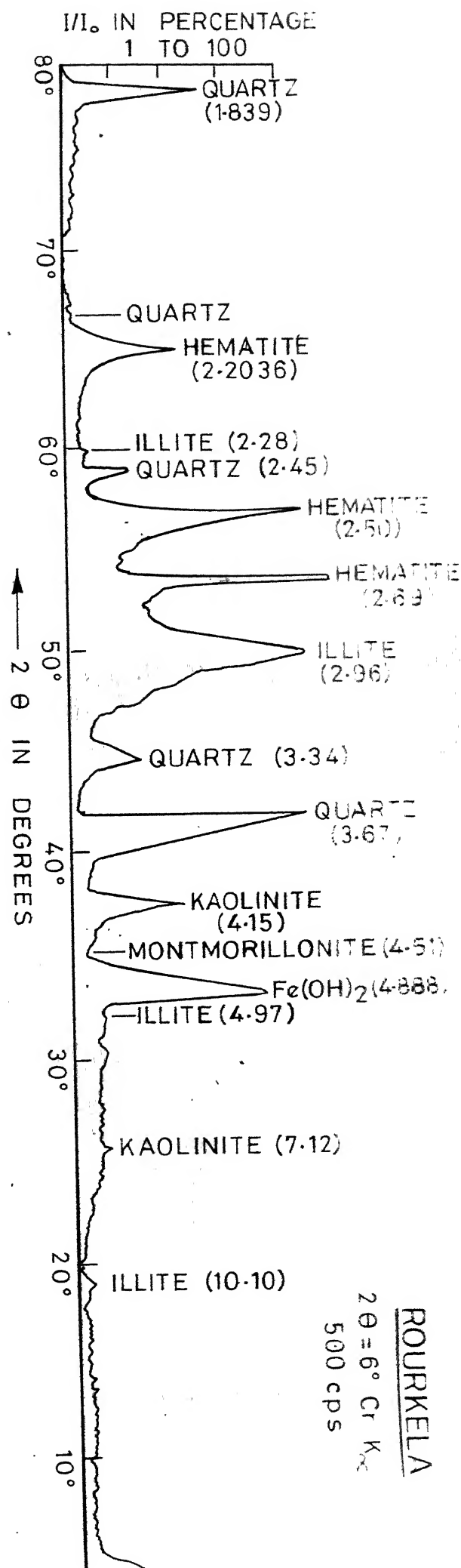
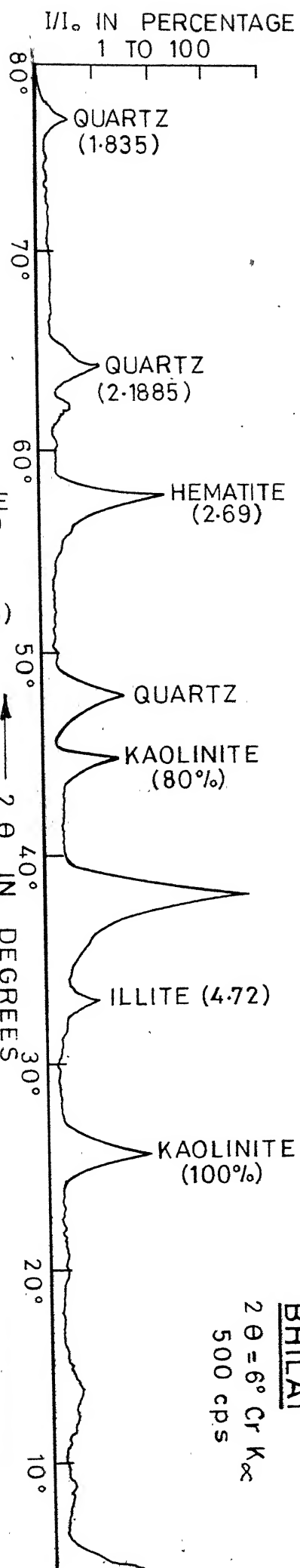
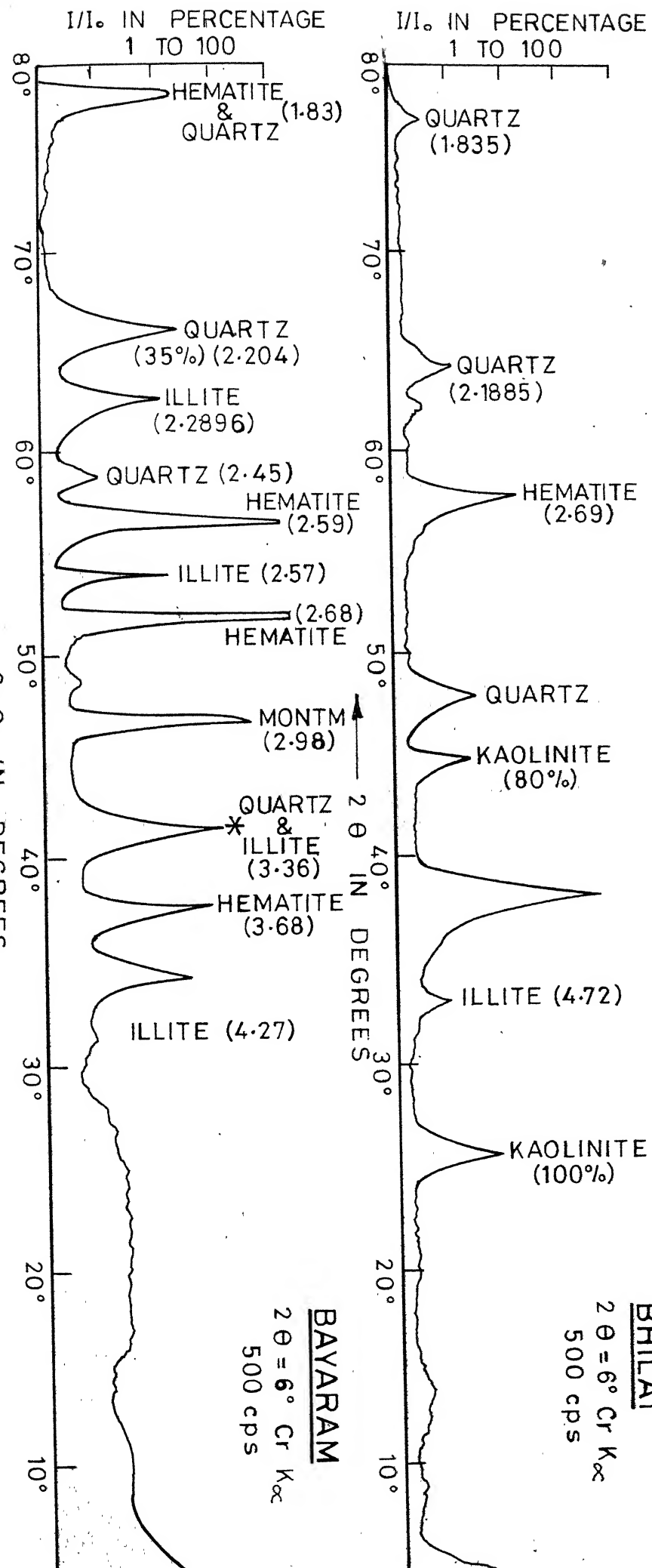
<u>Reference</u>	<u>Grain No.</u>	<u>Size in microns</u>	<u>Elemental Composition</u>
4(a)(b)	-	-	Si:Al:K:Mg = 5:3:1:1
4(c)(d)	-	-	Si:Al = 1:1
Micrograph	1	2.4x3.0	Al, Si, K
I	2	2.4x2.1	Al, Si
	3	4.2x2.1	Al (probably $Al_2O_3$ )
	4	1.9x1.1	Al, Si, K
II	1	6.2x4.3	Al, Si, K and Fe
	2	5.0x3.5	Fe (probably $Fe_2O_3$ )
	3	1.0x0.7	Al, Si, K
	4	0.9x0.8	Si (probably $SiO_2$ )
III	1	2.1x1.35	Al, Si, K
	2	1.9x1.4	" "
	3	1.5x1.1	Al, Si

---

TABLE IISize and Phase Composition of Typical Grains

Reference	Size in microns		Most likely minerals
603/604	Smallest	0.03x0.03	Quartz, Kaolinite, Illite
	Biggest	0.3x0.3	
605/606		0.6x0.3	Quartz, Montmorillonite, Diaspore
607/608	Smallest	0.03x0.03	Illite, Diaspore (?) Gibbsite (?)
	Biggest	0.67x0.32	
		0.64x0.37	
609/610		1.84x1.14	Montmorillonite, Illite Diaspore
611/612	Smallest	0.05x0.06	Illite, Gibbsite
	Biggest	1.67x0.6	

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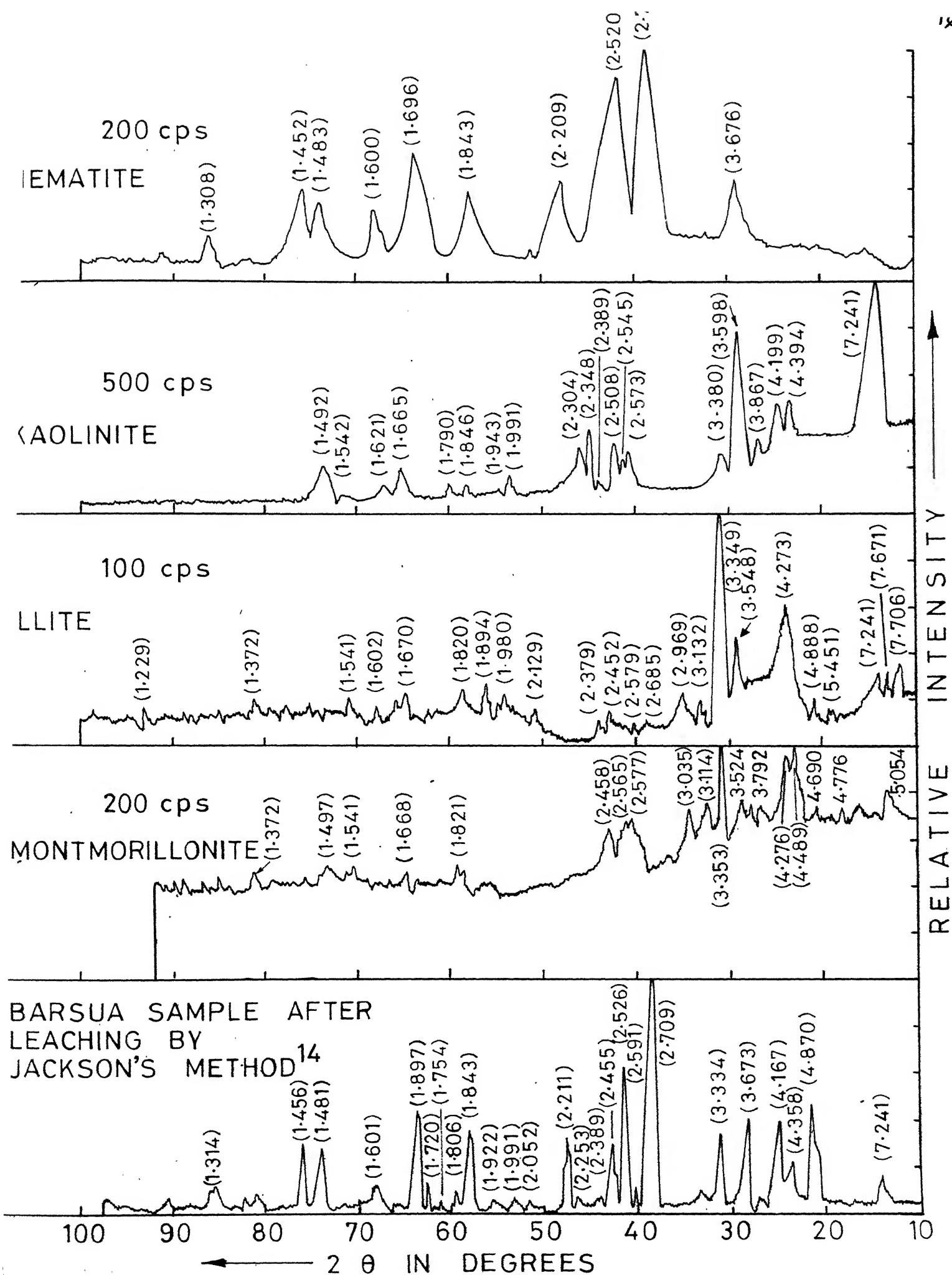


Fig. 3. X-RAY DIFFRACTOGRAM OF PURE AND UNKNOWN

## CHAPTER III

## CHARACTERISATION OF BHILAI IRON ORE

X-ray and electron microscopic investigations were made to ascertain the phase constituents in Bhilai iron ore. X-ray diffractogram (Fig. 2 given in the previous chapter) showed peaks for hematite, quartz, illite and kaolinite only.

13 electron diffraction patterns were taken: 7600436, 438, 440, ... 458 and 7600459. The corresponding micrographs were numbered one less (odd) i.e. 7600435, 437 ... etc. The camera length (L) for the diffraction patterns was 76 cms.  $\lambda = 0.37 \text{ \AA}$ , 'd' values were computed as  $L\lambda/R$  from the ring radii (R). The 'd' values computed were matched with the ASTM data for standard Al minerals. All minerals which corresponded to  $I/I_0$  less than 50 pct. were eliminated. Only those minerals which corresponded to 100 pct. intensity peaks and a large number of peak values were held as likely to be present.

The computed data are given in Table III. The most probable minerals arranged in decreasing order of occurrence frequency are: Sillimanite, Illite, Alunite, Hydrated aluminium silicate, Montmorillonite, Chloritoid, Kyanite, Corundum and kaolinite.

TABLE III  
SIZE AND COMPOSITION ON Al-CONTAINING GRAINS  
IN BHILAI ORE

Sl. No.	Micro-graph Plate No.	Particles size in microns	Prominent minerals from electron diffraction pattern	
1.	435	1.5 x 0.85	As in SAD pattern 436	Illite, Sillimanite
				Kyanite
2	437	2.10 x 2		Illite, Corundum, Kyanite, Sillimanite
3	439	0.6 x 0.5 1.5 x 0.5		Illite
4	441	1.1 x 0.25 1.4 x 0.35		Illite
5	443	0.85 x 0.4 0.3 x 0.45 0.15 x 0.15 0.6 x 0.25		Illite, Alunite, Hydrated aluminium silicate, calcium aluminite, chloritoid
6	445	1.75 x 1.5 2.25 x 2.25		Sillimanite
7	447	1.25 x 0.45 0.1 x 0.15 0.05 x 0.05		Sillimanite, Kaolinite
8	449	1.0 x 0.25 0.05 x 0.2		Illite, Montmorillonite
9	451	2 x 0.5		Illite, Kyanite
10	453	0.85 x 0.75 0.3 x 0.5		Illite
11	455	0.35 x 0.5 0.25 x 0.25 0.2 x 0.2 0.2 x 0.3 0.05 x 0.05 0.25 x 0.025		Sillimanite Alunite
12	457	0.35 x 0.15 0.9 x 0.5	As in SAD pattern 458	Sillimanite
13	-		Pattern 459	Sillimanite, Hydrated Aluminium Silicate

## CHAPTER IV

### SELECTIVE FLOCCULATION

Selective flocculation experiments were performed initially with Barsua Run of Mine ore and later with the 'slimes' generated in the Washing Plant. Flocculants used were Magnafloc 140 and 292 (cationic polyacrylamides) and Magnafloc 139 and 155 (anionics) supplied by Applied Colloids Limited, U.K. and also starch. Few more commercial polyacrylamide flocculants, which were tried and did not give satisfactory results, are also being referred to. Details about these flocculants are given in Appendix 'F'.  $\text{NaF}$ ,  $\text{Na}_2\text{SiO}_3$ ,  $\text{Na}_4\text{P}_2\text{O}_7$  were used as electrolytes in some experiments. Versicol W 13, a commercial dispersant, was used in some experiments.

Flocculation experiments were performed in a cylindrical vessel of 11 cm internal diameter and 24 cm, height i.e. of slightly more than 2 liter capacity. Samples were prepared such that the material settling in the vessel within 5 minutes (duration of a flocculation expt) was eliminated as too coarse, and ultra-fine slimes (not setting even in 2 hrs.) were also eliminated. The concentration of this sized sample (typically 1 to 30 microns) was kept around 4 gram per litre for flocculation experiments.

A typical experiment was initiated by mixing specific doses of dispersant, electrolyte and fine particles of iron ore or slime in the aqueous suspension measuring 2 liter.

Range of pH was kept around the normal equilibrium value i.e. 7-8. Some specific quantity of strong flocculant solution (typically 10 mg. dissolved in 2-3 drops of methanol and 100 c.c. distilled water to provide stock solution) was added and the system thoroughly agitated at high r.p.m. After 7 minutes, r.p.m. was reduced and flocs allowed to settle down within 2-5 minutes. Dispersants when used were added first as in stock solution to distilled water and stirred for 1 min., then slimes were added and stirred at high R.P.M. for 7 minutes to provide stable suspension. Then RPM was lowered, electrolyte and flocculant were added. 1 min. flocculation time resulted in the total time for experiment as 9 minutes. Flocculated and Non-flocculated portions were separated in single-stage experiments, for de-watering, drying and chemical assay.

In the multistage flocculation experiments, the flocculated portion in the 1st flocculation experiment (F1) was separated from the residue (NF1). F1 was re-dispersed and reflocculated using more dispersant and flocculant, giving F2 and NF2. F2 was similarly processed in the third stage to give F3 the flocculated part, and NF3. NF1, NF2 and NF3 were combined and subjected to the last or scavenging stage of flocculation yielding F4 and NF4. Different dosages of a stock flocculant solution were added in four specified stages. Thus, the ultimate products were three in number: F3, F4 the middling and NF4 the final non-flocculated fraction labelled as 'A' 'B' and 'C' respectively in Table VI etc.

## RESULTS

A large number of composite analysis of Barsua Run of Mine ore provided the analysis figures as Fe 58-64%,  $\text{SiO}_2$  1.5 to 2.0% and  $\text{Al}_2\text{O}_3$  4-6%. The 'slime' assayed Fe 52-55%,  $\text{SiO}_2$  5-7% and  $\text{Al}_2\text{O}_3$  8-12%. The composite R.O.M. sample also contained around 0.024% Cl, 0.66% K and 0.046% Mn.

Some results of single-stage flocculation experiments are given in Tables IV and V. Data on multiple-stage flocculation experiments are provided in Tables VI-X.

The following preliminary observations can be made:

1. Some alumina separation is achieved by cationic (imported) flocculants such as Magnafloc 292 (Table IV) and 140 (Table V and VI) for which lower dosage and multiple-stage flocculation seem advisable (Table VI)

2. Better beneficiation is achieved through the use of anionic (imported) flocculant Magnafloc 139 and use of dispersants (Table VII). A typical result gives a product with 52% iron recovery and only 20% of the total aluminium in the slime and assaying 3.5%  $\text{Al}_2\text{O}_3$ .

3. Expt. 3 in Table VIII using starch gives good separation of alumina but still  $\text{Al}_2\text{O}_3$  assay is not low enough and iron recovery is low.

4. Table IX shows that indigenous (Morarfloc) flocculants like A 10H and PG are not encouraging. A 40H was found to be

incapable of making stable flocs out of hematite fines.

Morafloc A30 and ATT may have some promise.

5. Commercial starch seems to have distinct promise. Expts. no. 6 and 7 in Table X indicate that it is possible to obtain a flocculated product assaying 88-91%  $\text{Fe}_2\text{O}_3$  and only 2.2-5.0%  $\text{Al}_2\text{O}_3$  corresponding to 77-82% recovery of  $\text{Fe}_2\text{O}_3$  and only 42-51% recovery of  $\text{Al}_2\text{O}_3$  in the beneficiated product. 160 ppm 'non-cooking' starch gives good results for commercial (expt 6) as well as chemical starch (expt. 4).

TABLE IV

## SINGLE STAGE FLOCCULATION OF BARSUA ROM BY SOME IMPORTED FLOCCULANTS

NO ADDITIVE SUCH AS DISPERSANT/ELECTROLYTE

C.....CATIONIC; A.....ANIONIC FLOCCULANT

F.....Floculated NF...Non-floculated fraction

Expt. No.	Flocculant used	Volume of 0.1 g/l flocculant solution used	pH	flocu- lated/ Floc(F)	in non- floc(NF)	recovery in <del>in</del> Al <sub>2</sub> O <sub>3</sub> SiO <sub>2</sub>		
1	Magnafloc 140(C)	0.6	7	19.5	6.96	9.16	76.2	72.6
2	"	0.6	8	28	6.6	6.8	79.6	81.0
3	"	0.6	8.8	22.4	6.4	6.8	78.4	77.5
4	Magnafloc 292(C)	1.5	7	40	8.8	4.4	57.6	72.1
5	"	1.0	8	27	4.3	4.5	75.2	73.1
6	"	1.0	9.2	18.5	4.0	4.4	62.3	57.8
7	HR 120 (C)	1.0	6.8	36	7.2	7.0	64.3	66.0
8	"	1.0	7.5	33	6.0	6.8	65.8	69.5
9	"	1.5	9.3	34	6.1	7.4	66.0	69.9
10	Magnafloc 155(A)	0.6	6.7	42	6.8	7.4	60.2	61.2
11	"	0.6	7.8	36	5.2	7.4	66.0	69.9
12	"	0.6	9.4	40	8.8	8.96	59.1	60.0



TABLE V

## SINGLE FLOCCULATION OF BARSUA SLIMES BY IMPORTED FLOCCULANTS

Pulp density 16g/2 lit. pH 7.9. 400 PPM Versicol W13 800 PPM NaF(used as 0.5M solution)

Expt. No.	Flocculant used	Quantity	% flocculated	% Al <sub>2</sub> O <sub>3</sub> Flocc(F)	in non-floc(NF)	% recovery in Al <sub>2</sub> O <sub>3</sub>	Non-floc SiO <sub>2</sub>
1	Magnafloc 140(C)	2 ml. of 0.2g/l	58.0	22.2	15.8	63.4	67.0
2	"	1 ml. of 0.5g/l	51.0	18.0	15.8	73.3	72.7
3	"	0.5 ml. of 0.8g/l	55.0	16.4	13.6	67.0	72.3
4	Magnafloc 292(C)	2 ml. of 0.2g/l	20.1	15.6	14.6	78.6	82.7
5	"	1.5 ml of 0.5g/l	33.0	16.8	17.2	70.9	68.4
6	"	0.9 ml. of 0.8g/l	28.5	19.2	19.2	64.2	73.0
7	Magnafloc HR 120(C)	1.8 ml. of 0.2g/l	30.1	17.8	15.2	65.9	63.6
8	"	1.5 ml. of 0.5g/l	32.9	15.2	15.6	67.0	61.8
9	"	0.75ml. of 0.8g/l	28.8	16.8	16.0	69.5	70.2
10	Magnafloc 139(A)	1.2 ml. of 0.2g/l	26.6	19.2	19.8	73.8	73.5
11	"	1.5 ml. of 0.5g/l	21.5	21.2	21.2	78.8	80.0
12	"	0.75 ml. of 0.8g/l	30.7	16.0	16.4	68.9	66.4

TABLE VI

MULTIPLE STAGE FLOCCULATION OF BARSUA SLIME BY MAGNAFLOC 140(C)  
 SCHEME AND SYMBOLS 'A','B','C' EXPLAINED IN THE TEXT  
 INITIAL PULP CONCENTRATION ~ 16g/2 lit. 400 PPM VERSICOL W13 800 PPM NaF  
 (A TYPICAL COMPOSITE ANALYSIS OF SLIME: 14.8%  $Al_2O_3$ , 12.2  $SiO_2$  and 69.8  $Fe_2O_3$ )

Expt. No.	Flocculant Dosages(ml.)	Frac-tions	Wt. %	A S S A Y %			RECOVERY %		
				$Fe_2O_3$	$SiO_2$	$Al_2O_3$	$Fe_2O_3$	$SiO_2$	$Al_2O_3$
1	2.5, 2.0, 1.5 and 2.5 of 0.2 g/l solution in 4 steps	A	16.1	65.6	8.4	26.0	15.8	13.4	24.1
		B	69.6	70.4	10.8	15.6	72.9	77.8	63.6
		C	10.9	69.6	7.8	19.2	11.3	8.8	12.4
2	3, 2, 1 and 3 of 0.5 g/l	A	27.2	67.2	12.2	10.0	27.7	29.5	20.5
		B	56.3	67.2	10.7	14.4	57.6	54.8	61.2
		C	14.8	65.6	11.8	16.4	14.8	15.7	18.3
3	3, 2, 1 and 3 of 0.6 g/l	A	37.1	69.6	11.0	16.4	37.7	40.5	32.9
		B	41.97	68.8	8.6	22.0	42.1	35.7	49.9
		C	20.3	68.4	11.8	15.6	20.2	23.7	17.1
4	1, 0.8, 0.3 and 1.0 of 0.8 g/l	A	23.6	65.6	12.0	14.8	23.2	23.8	24.4
		B	40.9	70.4	12.4	14.96	43.1	42.9	42.7
		C	32.1	70.0	12.2	14.68	33.7	33.2	32.9

TABLE VII

## MULTIPLE STAGE FLOCCULATION OF SLIME BY MAGNAFLOC 139(A)

PULP DENSITY 16 g./2 lit. 800 PPM NaF

Expt. No.	Dispersant used	Dosages of 0.6 g/l flocculant soln. (ml.)	Frac- tions	Wt %	A S S A Y			RECOVERY %	
					Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub> Al <sub>2</sub> O <sub>3</sub>
1.	Versicol WL3 400 PPM	2, 3, 0.5 and 1.5	A	51.2	68.8	11.8	17.6	52.6	53.3 48.5
			B	23.8	68.8	10.8	18.4	24.4	22.7 23.5
			C	23.3	66.4	11.7	22.4	23.0	24.0 27.1
2	50 PPM Versicol WL3 400 PPM Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	2, 1, 0.5 and 1.5	A	20.0	68.8	6.3	3.4	20.8	13.1 9.1
			B	56.7	67.2	12.4	5.6	57.6	73.1 42.5
			C	21.4	66.4	6.2	16.8	21.3	13.9 48.3
3	50 PPM Versicol WL3 400 PPM Na <sub>2</sub> SiO <sub>3</sub>	2, 1, 0.5 and 1.5	A	50.8	76.2	11.8	3.6	51.9	53.5 19.5
			B	22.3	70.4	11.2	17.8	23.9	22.3 42.3
			C	25.3	62.8	10.7	14.2	24.2	24.2 38.2

TABLE VIII

SOME TYPICAL FLOCCULATION EXPERIMENTS WITH MAGNAFLOC 139(A) AND  
INDIGENOUS FLOCCULANTS. 800 PPM NaF

Expt. No.	Dosages of Dispersants and Flocculants	Frac- tions	Wt %	A S A Y %			RECOVERY %		
				Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>
1.	400 PPM Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	A	3.6	60.7	11.8	8.6	3.7	4.8	2.0
	50 PPM Versicol WL3,2,	B	75.1	70.4	9.9	18.5	89.5	83.5	91.2
	2, 10.5 and 1.5 ml. of 10 g/l. M 139	C	6.9	58.4	15.1	14.8	6.8	11.2	6.7
2.	Dispersants as above	A	32.0	64.4	10.5	17.9	39.3	31.0	35.4
	20 ml. of 10g/l. starch soln. (single stage flocculation)	B	54.6	57.6	13.5	19.3	60.7	69.0	64.6
3.	Same as above except Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	A	30.7	68.8	10.5	14.8	33.6	28.8	24.6
	replaced by Na <sub>2</sub> SiO <sub>3</sub> and starch	B	54.8	66.8	11.8	19.9	58.4	57.8	59.5
	soln. 3, 2, 5 and 6 ml. (multiple stage)	C	9.1	55.5	16.6	32.3	8.0	13.4	15.9
4.	Dispersants as in Expt. 1	A	33.1	68.8	11.1	14.0	35.1	33.3	33.4
	Morarfloc AlOO flocculant	B	24.3	68.8	11.4	15.8	25.8	25.0	27.6
	10 g/l : 2, 1, 0.5 and 1.5 ml	C	38.1	66.8	12.1	14.3	39.2	41.7	39.1
5.	Dispersants as in 3	A	52.9	68.8	10.3	12.9	54.7	52.4	55.0
	Flocculant as in 4:	B	31.3	66.4	10.7	11.5	31.2	31.9	28.9
	4, 3, 2 and 4 ml	C	14.3	66.0	11.4	14.1	14.2	15.6	16.2

TABLE IX

## MULTI-STAGE EXPERIMENTS WITH INDIGENOUS FLOCCULANTS AND BARSUA SLIMES

Electrolyte used: NaF solution 0.1M 0.8, 0.6, 0.4 and 0.8 c.c. in 4 stages										
Dispersant : Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub> " 185PPM 8, 6, 4 and 8 c.c.										
Versicol W13 " 1000 PPM 2, 1, 0.5 and 2 c.c.										
Floculant " 1000 PPM 2, 1, 0.5 and 2 c.c.										
Expt. No.	Floculant	Fractions	Wt %	A S S A Y %			R E C O V E R Y %			
				Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	
1	Morarfloc A 10 H	A B C	73.1 8.0 13.5	67.8 65.0 62.0	11.3 11.4 11.5	14.8 15.9 15.5	78.5 8.2 13.2	77.0 8.5 14.5	76.3 9.0 14.7	
2	A 40 H	A B C	71.5 13.0 5.8	67.5 78.8 55.7	11.3 7.7 15.8	13.3 9.8 18.5	78.1 16.6 5.2	80.7 10.0 9.2	80.2 10.7 10.0	
3	A 30	A B C	79.2 7.7 9.7	71.5 60.0 54.0	9.3 12.9 15.4	14.9 18.0 19.2	85.1 6.9 7.9	74.9 10.0 15.1	78.4 9.2 12.4	
4	PG	A B C	62.2 6.8 7.0	68.0 63.3 59.6	11.7 13.7 15.0	13.4 13.7 16.4	86.9 6.7 6.5	83.0 8.0 9.0	84.1 7.1 8.8	
5	Starch (0.1M Na <sub>2</sub> SiO <sub>3</sub> used instead of Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub> )	A B C	83.3 4.6 7.4	69.0 65.7 63.0	9.5 8.9 11.6	15.6 18.0 19.6	88.3 4.6 7.1	86.2 4.5 9.3	85.3 5.3 9.4	
6	A - TT	A B C	60.0 16.4 16.0	70.0 68.0 65.3	10.6 11.9 13.2	16.3 16.2 20.6	66.0 17.6 16.4	61.1 18.7 20.2	62.3 16.9 20.9	

TABLE X

## MULTISTAGE FLOCCULATION OF BARSUA SLIME WITH STARCH SOLUTIONS

NaF, 0.1 M  $\text{Na}_2\text{SiO}_3$ , Versicol WL3, Flocculant Dosages as in Table IX

Non-cooking/Aging: Chemical or commercial starch was made into a paste, heated at  $60^\circ\text{C}$  for half an hour and aged for 2 days

Cooking: Solution was heated at  $95^\circ\text{C}$  for  $\frac{1}{2}$  an hour. No ageing, freshly used.

Solution strength 1000 PPM or 160 PPM

Expt. No.	Flocculant	Strength and Preparation	Fract-ions	Wt. %	A S S A Y			RECOVERY %		
					$\text{Fe}_2\text{O}_3$	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$
1	Chemical Starch	1000 PPM cooking	A	76.0	92.3	2.2	5.1	81.6	52.8	62.7
			B	13.0	88.7	4.0	7.1	13.5	13.5	15.1
			C	7.0	60.1	18.3	19.6	4.9	33.6	22.2
2	"	160 PPM cooking	A	81.9	85.3	5.7	9.0	78.4	65.8	70.4
			B	1.3	89.1	3.5	7.4	13.4	6.6	9.6
			C	11.3	64.3	17.3	18.4	8.1	27.6	20.0
3	"	1000 PPM Non-cooking	A	80.8	91.5	1.7	6.8	78.5	34.3	61.4
			B	1.3	88.8	3.4	7.8	12.2	11.2	11.4
			C	13.2	65.3	16.4	18.3	9.2	54.6	27.2
4	"	160 PPM Non-cooking	A	82.0	90.9	4.5	4.6	84.6	68.3	60.2
			B	9.4	88.0	4.0	8.0	9.4	7.0	12.2
			C	8.3	63.8	16.1	20.1	6.0	24.7	26.9

Contd.

(Table X Contd.)

Expt. No.	Floculant	Strength and preparation	Fract-ions	Wt	A S A Y			RECOVERY		
					Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>
5	Commercial starch	1000 PPM Non-cooking	A	80.1	89.4	2.96	7.6	<u>83.4</u>	67.4	75.9
			B	10.5	90.8	2.2	7.0	11.1	6.6	9.2
			C	6.9	69.3	13.3	17.4	5.5	26.0	14.9
6	"	160 PPM Non-cooking	A	70.2	91.8	3.2	<u>4.96</u>	<u>81.8</u>	39.3	<u>41.7</u>
			B	7.0	81.1	9.8	9.1	7.2	11.9	7.6
			C	15.7	55.0	18.0	27.0	10.96	48.8	50.7
7	"	1000 PPM cooking	A	63.7	88.2	9.6	<u>2.2</u>	<u>77.3</u>	66.8	<u>50.9</u>
			B	4.7	75.1	11.5	13.4	4.9	5.9	23.4
			C	16.2	80.2	15.5	4.3	17.8	27.4	25.7
8	"	160 PPM cooking	A	69.5	85.7	8.8	5.5	77.2	67.6	50.5
			B	6.6	75.5	10.3	13.3	6.5	7.5	11.6
			C	17.7	71.1	12.7	16.2	16.3	24.8	37.9

## CHAPTER V

### SETTLING CHARACTERISTICS OF PURE MINERALS

Mineral particles must be selectively or non-selectively dispersed to achieve the desired degree of selective flocculation. In order to study stability of suspensions, column studies<sup>19</sup> were undertaken. The studies were carried out on pure minerals of Hematite, Illite, Kaolinite and Montmorillonite. In order to avoid size-effect of minerals on their settling behavior, only closely sized particles were considered. The range of the particle size was kept in between -20 and +5 microns (as this size range was more suitable for flocculation studies). Using the Stokes equation, settling times were calculated for -20 and +5 micron size particles. Specific gravity values of the mineral were found by using petroleum ether (sp. gr. 0.67) as medium of liquid. These values were used in calculating terminal velocity in the Stokes equation.

Settling experiments were carried out in a cylindrical column of 50 cm height, 10 cms dia and having 5 outlets at equal intervals of 10 cms. outlet dia being 5 mm. Suspension was thoroughly stirred and taken into column and allowed for settling at different intervals of time. Samples were taken out at different timings, and concentration measured. Concentration of suspension at different depths and at different



timings were obtained and used to draw graph between concentration and depths. The curve thus obtained shows the settling characteristics of a suspension. For a good dispersed system we should get a vertical straight line as the plot. Settling column was prepared with the help of some details given in a previous publication<sup>19</sup>.

Stability studies of pure minerals were carried out with dispersants such as sodium pyrophosphate ( $\text{Na}_4\text{P}_2\text{O}_7$ ), sodium silicate ( $\text{Na}_2\text{SiO}_3$ ), Versicol-W13 and combination. The effect of dispersant on the mineral system was found out. Stability numbers (percentages) were calculated with the help of curves which give an idea about stability at particular time (t) and at particular height (h). These numbers are comparable and help us to choose between dispersants and systems.

#### PROCEDURE

In order to avoid interference of size effect on settling behavior of minerals, only closely sized particles were considered. The range of particle size was kept at  $-20 + 5$  microns. A settling column of 100 cms height 10 cm dia and with an outlet dia of 1 cm was used. Using the Stokes equation, the time required for particle to settle through 100 cms column was calculated.

$$\begin{aligned}\text{Volume of the column} &= \pi r^2 h = 3.14 \times 5^2 \times 100 \\ &= 7850 \text{ cc}\end{aligned}$$

$$\text{Stokes equation} = V_t = \frac{1}{18} \frac{d^2 g}{\mu} (\rho_s - \rho_o)$$

where  $V_t$  = terminal velocity

$d$  = dia of particle size

$g$  = gravity

$\mu$  = coefficient of viscosity

$\rho_s$  and  $\rho_o$  are specific gravity of solid and fluid respectively.  $\rho_s$  values for,

Hematite = 5.68, Kaolinite = 2.78.

Illite = 2.425, Montmorillonite = 2.64

$$V_t = \frac{1}{18} \times \frac{4 \times 10^{-6} (5.68 - 1.00) \times 981}{.01} = 10.2 \text{ cms/sec.}$$

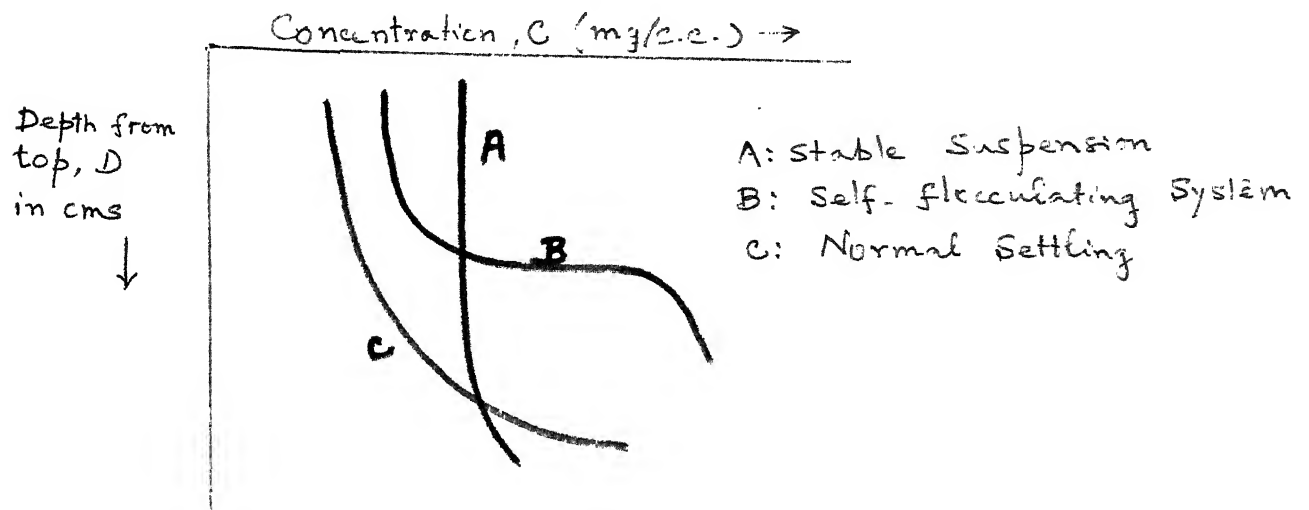
$$t, \text{ time} = \frac{S}{V_t} = \frac{100}{10.2} \approx 10 \text{ sec. for } + 20 \text{ micron size particle hematite}$$

$$\text{and for } d = 5 \text{ microns, } t = (4)^2 \times 10 = 160 \text{ sec.} = 2'30''$$

Hence particles settling in between 10 sec. and 2'30'' were collected for the experimental purpose. Simillar calculations were done for kaolinite, Illite and montmorillonite. Thick slurry was added to the water column and the particles allowed to settle in the calculated timings. Table XI shows the fractions obtained (weights). Experiment was repeated till sufficient amount of sample was obtained. The samples were tested under microscope and size range was confirmed qualitatively.

The variables in conducting the column settling tests are column size, no. of sampling ports, the time versus depth curves and the initial suspended solid concentration of the test<sup>19</sup>. Here the column dimension and no. of sampling ports were fixed. Particle concentration can be varied from 1 to 10 pct. In our case, it was fixed as 1 pct.

1 pct. suspension was thoroughly stirred and taken into a column and allowed for settling at different timings, and concentration was measured gravimetrically. Curves were drawn with concentration on x axis and depth on y-axis.



For a stable suspension  $\frac{d(D)}{d(C)}$  should be infinity. The depth at which  $\frac{d(D)}{d(C)}$  falls drastically, indicates the zone for pronounced settling. If it occurs at larger depth, then it is normal settling and if it occurs at smaller depths than it

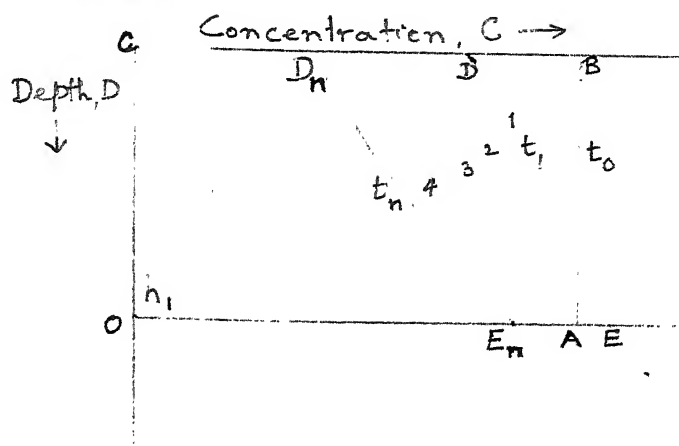
indicates self-flocculating characteristics<sup>19</sup>. In order to take care of all the above factors, the area under dispersed (ideal) system and the actual one were compared. The percentages thus evolved indicate stability numbers. Results are tabulated in Table XII.

39.25 gms of material were taken in 3925 cc i.e. 10 mg/cc concentrated pulp was prepared. While preparing the pulp, care was taken to avoid floc formation. The material was taken in one litre beaker and water was added drop by drop in the initial stages, and stirred with a glass rod to avoid floc formation and thus slurry was prepared. This was diluted to a concentration of 10 mg/cc. Dispersant was added (as 0.1 M stock soln. in case of  $\text{Na}_4\text{P}_2\text{O}_7$  and  $\text{Na}_2\text{SiO}_3$  and as 1 pct. by vol. with Versicol-W13) and conditioned for 1 hr.

The above pulp was taken into the settling column of 50 cm ht. and 10 cm dia. and allowed to settle. Samples of 40 cc were taken at different timings and at different depths. In our case, timings were kept at 1, 5, 15, 30 and 60 minutes, and depths at 10, 20, 30, 40 and 50 cms. Concentration of sample was found by gravimetric method i.e. the sample was dried on a heater or in an oven, and residual weights were noted.

As the time increases the stability of the pulp decreases. Hence the concentration of sample at <sup>smaller</sup> ~~lower~~ depth decreases.

Curves were drawn taking concentration on x-coordinate and depth on y-coordinate.



For a well dispersed system the concentration of sample should be same at all depths i.e. it should be a straight line parallel to y-axis. Curves 1, 2, 3, 4 correspond to data at different times. The area covered by the curve (at particular height) gives the amount of material present in the pulp. Hence a comparison between areas covered at ideal condition (dispersed system) and with the area under actual curve indicates stability at the height (h) and at that particular time (t).

At depth  $h_1$  and at time  $t_1$  stability of the suspension is  $S_{h_1 t_1} = \frac{\text{Area under OEDC}}{\text{Area under OABC}} \times 100$  which indicates stability at depth  $h_1$  and at time  $t_1$ . In all cases  $\frac{S_{h, t_0}}{S_{h, t_1}}$  is taken as 100. Similar calculations were made for other curves. Results are provided in Tables XI to XIII and Figs. 5-10.

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RESULTS

TABLE XI

PREPARATION OF MINERAL FINES

Sl. No.	Mineral	Density (sp.gr.)	Size in microns	Settling time in min(')sec('')	Amount   pct. by wt-	
					(for one run) in gms	in pct.
1.	Hematite	5.68	+ 20	10 sec	20.35	26.4
	"	"	- 20 + 5	2'30"	28.24	36.65
	"	"	- 5 + 1	80'-90'	25.77	33.4
2.	Illite	2.425	+ 20	31.8"	14.05	18.3
	"	"	- 20 + 5	8'31"	19.289	25.1
	"	"	- 5 + 1	212'40"	37.896	49.25
3.	Kaolinite	2.78	- 20	25.8"	14.115	18.4
	"	"	- 20 + 5	6'59"	41.831	54.3
	"	"	- 5 + 1	172'	16.041	20.4
4.	Montmori- llonite	2.64	+ 20	28"	17.05	22.2
	"	"	- 20 + 5	7'28"	17.8	23.1
	"	"	+ 1	187'	25.3	32.9

TABLE XII

STABILITY NUMBER OF MINERALS

Sl. No.	Stability No.	Hematite (H) pct.	Kaolinite (K) pct.	Illite (I) pct.	Montmorillonite (M) pct.
1	$S_{30}t_1$	30.7	84.2	73.3	73.3
2	$S_{30}t_5$	12.05	85.5	62.4	49.9
3	$S_{30}t_{30}$	7.06	41.7	29.05	-
4	$S_{40}t_1$	24.35	95.4	86.0	75.0
5	$S_{40}t_5$	15.28	89.0	69.25	57.9
6	$S_{40}t_{30}$	14.37	45.3	41.75	-

$S_{ht}$  defined before. h in cms t in minute.

TABLE XIII

## STABILITY NUMBER OF MINERALS IN DISPERSANT SOLUTIONS

Stabi- lity No.	HEMATITE (H)		MONTMORILLONITE (M)				KAOLINITE (K)				ILLITE (I)				
	S.P.	S.S	S.P.&S.S	V-W13	S.P.	S.S	S.S.&S.P.	V-W13	S.P	S.S	S.S & S.P.	V-W13	S.P	S.S	S.S & S.P.
S <sub>30</sub> <sup>t1</sup>	50.5	53.9	60.0	84.16	80.55	56.1	74.2	98.64	88.9	92.8	83.34	99.9	72.2	66.7	62.8
S <sub>30</sub> <sup>t5</sup>	22.23	58.8	34.4	28.3	73.5	53.4	38.8	93.9	75.2	63.4	74.25	82.8	52.6	53.6	70.4
S <sub>30</sub> <sup>t30</sup>	10.0	6.67	10.0	10.0	31.9	8.33	9.8	83.9	44.4	11.69	27.75	25.1	34.4	19.47	30.0
S <sub>40</sub> <sup>t1</sup>	70.2	61.5	64.5	91.25	83.52	69.9	72.2	99.0	83.35	94.09	87.0	99.9	76.25	72.9	70.0
S <sub>40</sub> <sup>t5</sup>	26.46	54.3	46.25	31.05	73.25	47.9	44.43	95.5	79.6	69.03	78.6	92.08	59.4	56.4	43.1
S <sub>40</sub> <sup>t30</sup>	10.0	7.5	10.0	10.8	27.5	7.50	10.83	90.4	51.54	13.5	62.75	23.2	61.2	22.7	34.9

Note:

S<sub>30</sub><sup>t1</sup> Indicates stability at a depth of 30 cms and at 1 minute, etc.

S.P. Sodium pyro phosphate ( $\text{Na}_4\text{P}_2\text{O}_7$ )

S.P. & Added as 0.1M Stock Soln.  
S.S. 16 ml. in 4000 cc.

S.S. Sodium silicate ( $\text{Na}_2\text{SiO}_3$ )

V-W13 Added as 1% (by vol.) soln.

S.P. & S.S. Sodium Phosphate & Sodium silicate

1.6 ml

V-W13 Versicol-W13

Final concentrations were 90, 48 and 4 PPM for SP, SS and VW13



## EFFECT OF DISPERSANT CONCENTRATION ON STABILITY OF SUSPENSION

Some experiments were carried out to find the effect of dispersant concentration on the settling characteristics of suspensions. The materials under consideration were Hematite and Montmorillonite (size range -  $20 + 5$  microns). In this case, only dosage of dispersant was varied and the amount of material in suspension at  $2/3$ rd height was measured.

A small settling column (cylindrical jar) with one sampling port at  $1/3$ rd distance from the base was used. The dimensions of the small jar was 3 cm dia, 16.5 cm height (total). Height of the solution (100 cc) in cylinder was 11.5 cms, and sampling port height from the base was kept at 3.5 cms.

### PROCEDURE

6 gms of material was taken in 600 cc of water and stirred well for 30 mts. First, dispersant was added into the settling column. Then pulp of 10 mg/cc concentration was added to it i.e. Addition of pulp was preceded by addition of dispersant dosage. It was shaken for one minute and allowed for settling, at definite interval of time. (In our case it was 15 minutes for Montmorillonite, and 5 minutes for Hematite). Dosages of dispersant employed were 0, 50, 100, 200, 350 and 500 ppm. Unsettled fraction (upto the sampling port level) was taken out from the sampling part, dried and weighed, 5-10 pct. handling losses were involved in conducting the test.

A graph was drawn between <sup>fraction of</sup> unsettled portion and dispersant concentration. Amount of material in suspension was taken on y-axis, Dosage of dispersant was taken on x-axis. The experiments were carried out with sodium silicate and sodium pyrophosphate. Dispersant were used as 0.1M stock solution. Results are tabulated in Table No. XIV and XV, and ~~(#)~~ and also shown in Figs. 11 and 12.

## RESULTS

TABLE XIV

### DISPERSION WITH SODIUM SILICATE ( $\text{Na}_2\text{SiO}_3$ )

Sl. No.	Dosage of dispersant in ppm	Montmorillonite(in mgs)			Hematite (in mgs)		
		Unsettled	Settled	Total	Unsettled	Settled	Total
1	0	162.5	769.8	932.3	127.5	792.5	920.0
2	50	298.6	640.4	939.0	120.2	779.8	900.0
3	100	324.2	648.5	973.0	139.3	760.2	899.5
4	200	344.2	549.6	893.8	169.0	741.0	910.0
5	350	346.2	537.1	883.3	202.0	688.0	890.0
6	500	352.8	500.5	853.3	229.4	650.6	880.0

TABLE XV

DISPERSION WITH SODIUM PYROPHOSPHATE

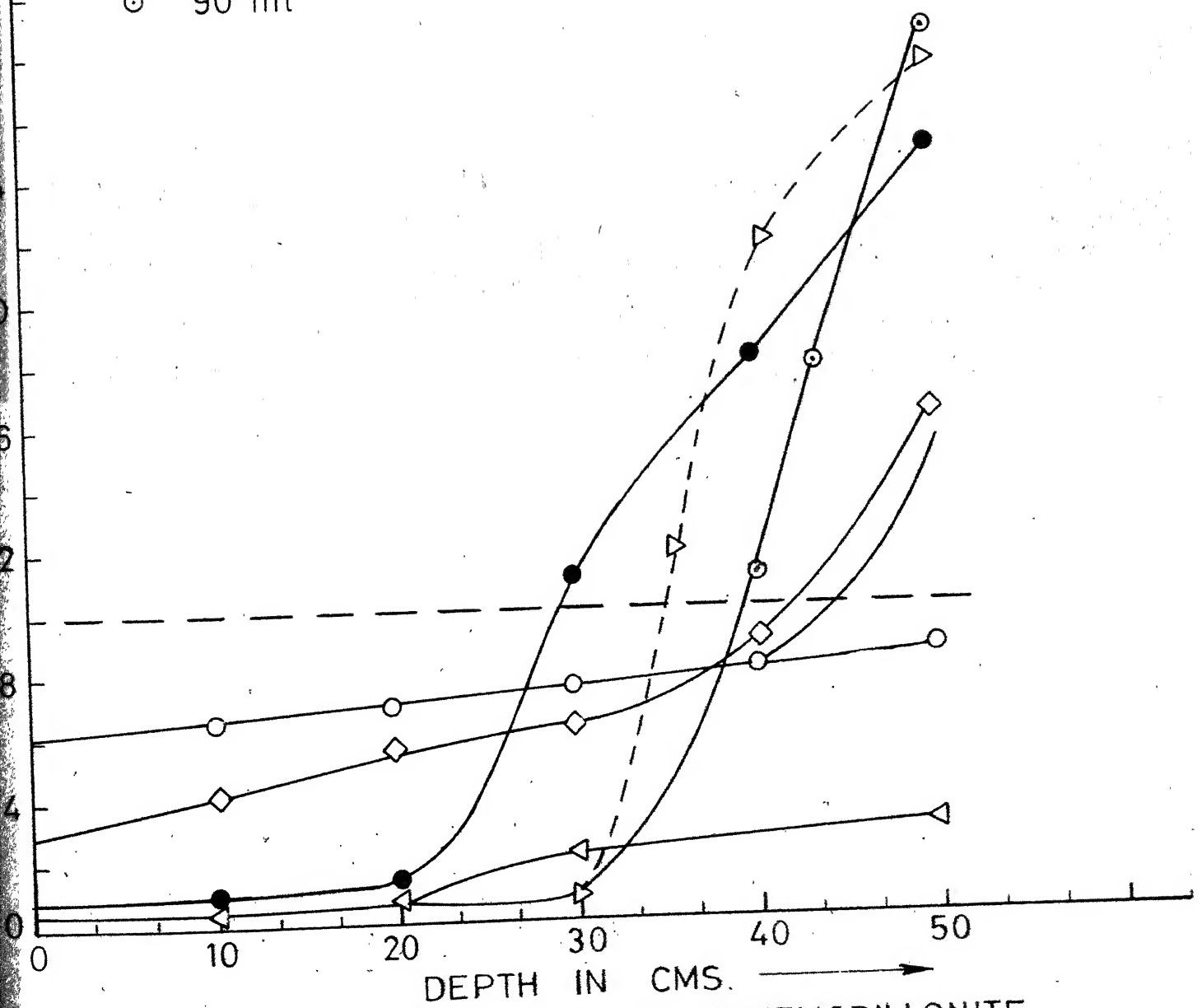
Sl. No.	Dosage of dispersant in ppm	<u>Montmorillonite(in mgs)</u>			<u>Hematite (in mgs)</u>		
		Unsettled	Settled	Total	Unsettled	Settled	Total
1	0	162.5	769.8	932.3	125.0	825.0	950.0
2	50	324.0	632.0	956.0	122.3	800.0	922.3
3	100	365.0	600.0	965.0	135.0	798.0	933.0
4	200	377.0	598.0	975.0	170.0	780.0	950.0
5	350	443.0	502.0	945.0	194.0	779.0	973.0
6	500	442.6	501.0	943.6	-	-	-

CONCLUSIONS

In most cases, kaolinite was found to be the best dispersant mineral and Hematite the poorest. This could be due to density effect, charge effect or both. For Montmorillonite and Illite,  $\text{Na}_2\text{P}_4\text{O}_7$  was found to be a better dispersant compared to  $\text{Na}_2\text{SiO}_3$ . Such a superiority was not established for kaolinite and hematite. Combination of  $\text{Na}_4\text{P}_2\text{O}_7$  and  $\text{Na}_2\text{SiO}_3$  may serve the purpose equally well. Versicol W13 has some effect on Montmorillonite. Typical dispersant concentrations may be chosen as 50-100 PPM on the lower side and 300-400 PPM on the higher side (vide Fig. 11).

## LEGEND

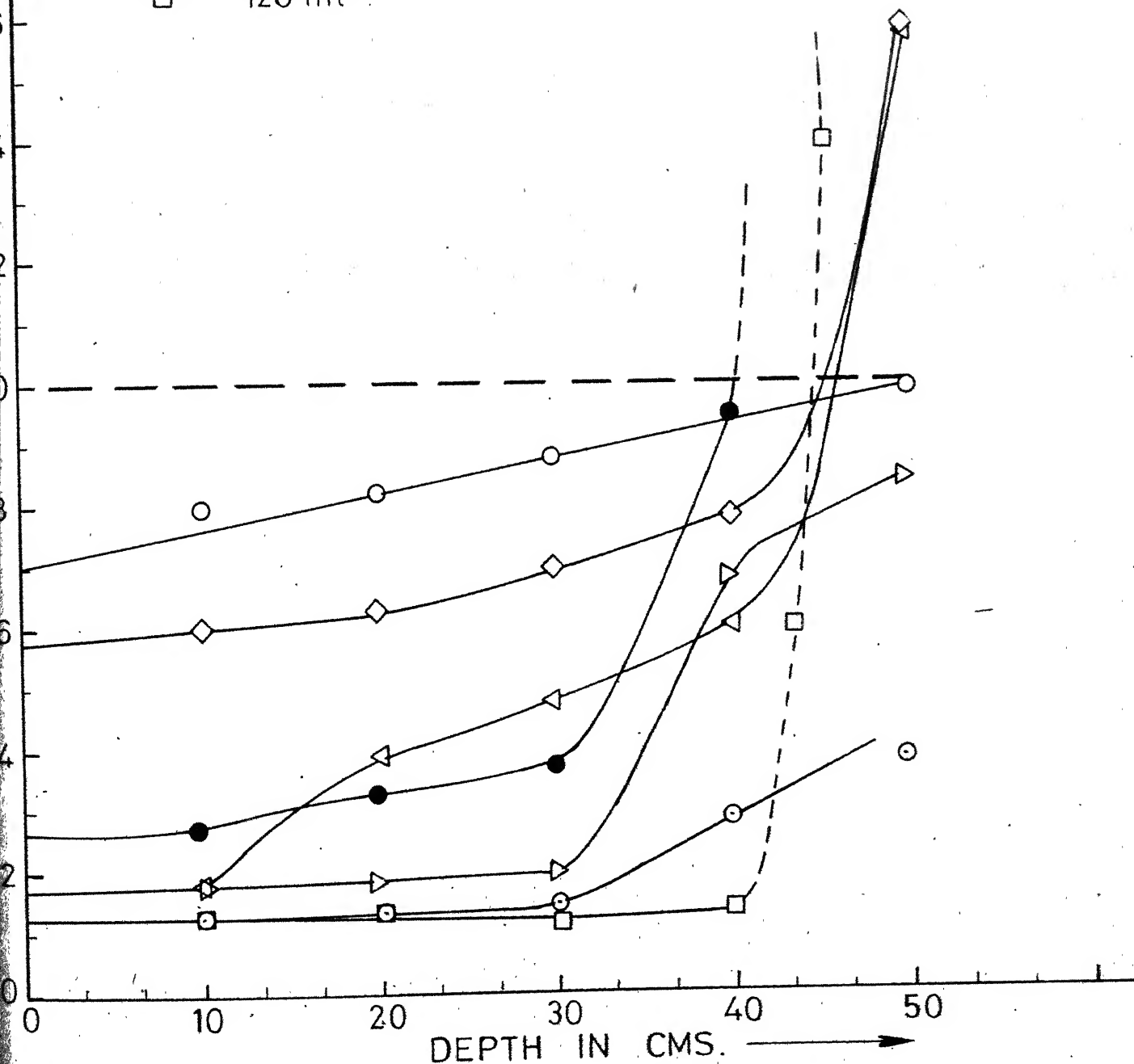
- 0 mt  
 ○ 1 mt  
 ◇ 5 mt  
 ● 15 mt  
 △ 30 mt  
 ▽ 60 mt  
 ⊙ 90 mt



5 SETTLING CHARACTERISTICS OF MONTMORILLONITE WITHOUT DISPERSANT.

## LEGEND

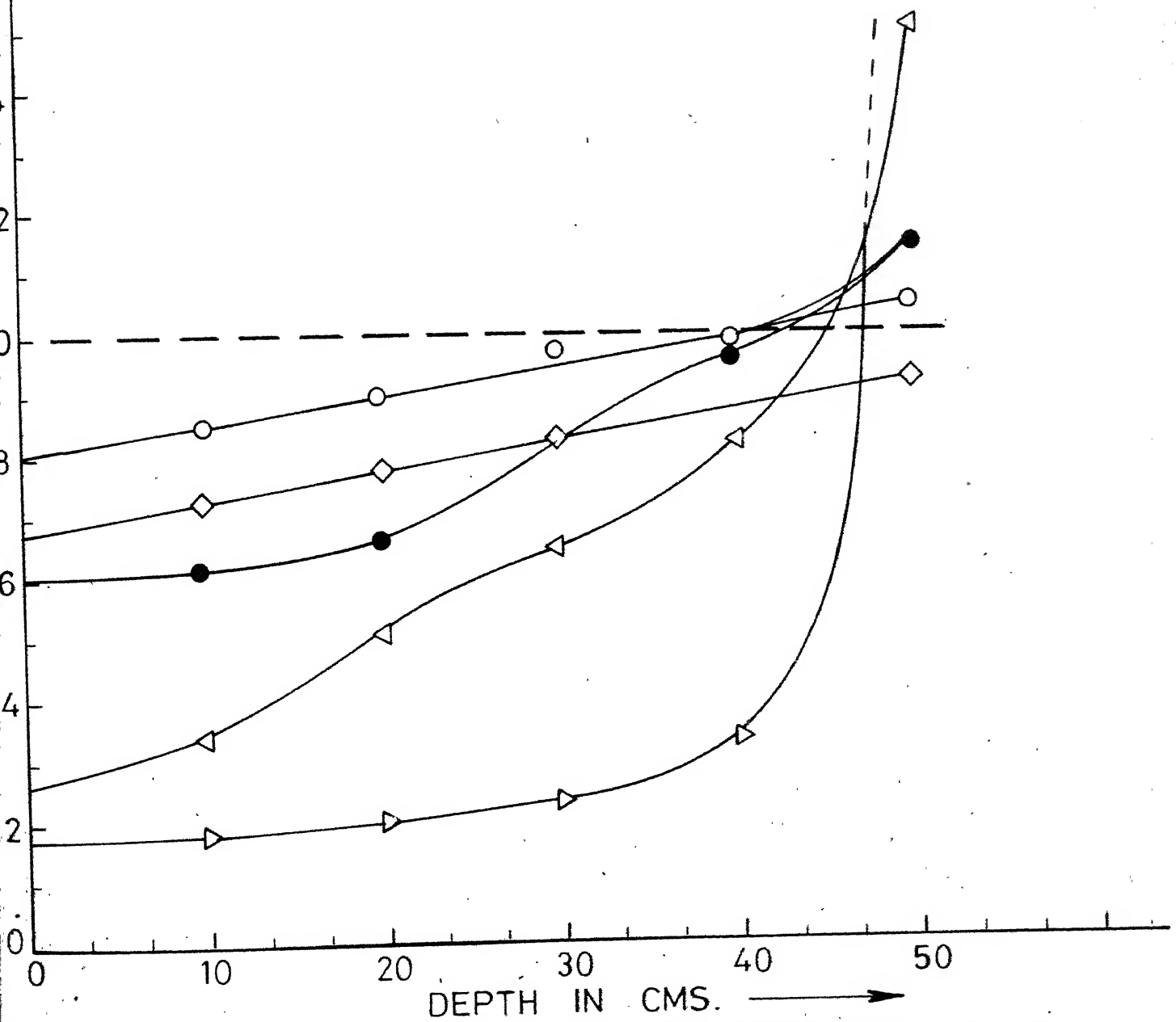
- 0 mt  
 ○ — 1 mt  
 ◇ — 5 mt  
 ● — 15 mt  
 △ — 30 mt  
 ▽ — 60 mt  
 ⊙ — 90 mt  
 □ — 120 mt



6. SETTLING CHARACTERISTICS OF MONTMORILLONITE WITH DISPERSANT ( $\text{Na}_4\text{P}_2\text{O}_7$ ).

## LEGEND

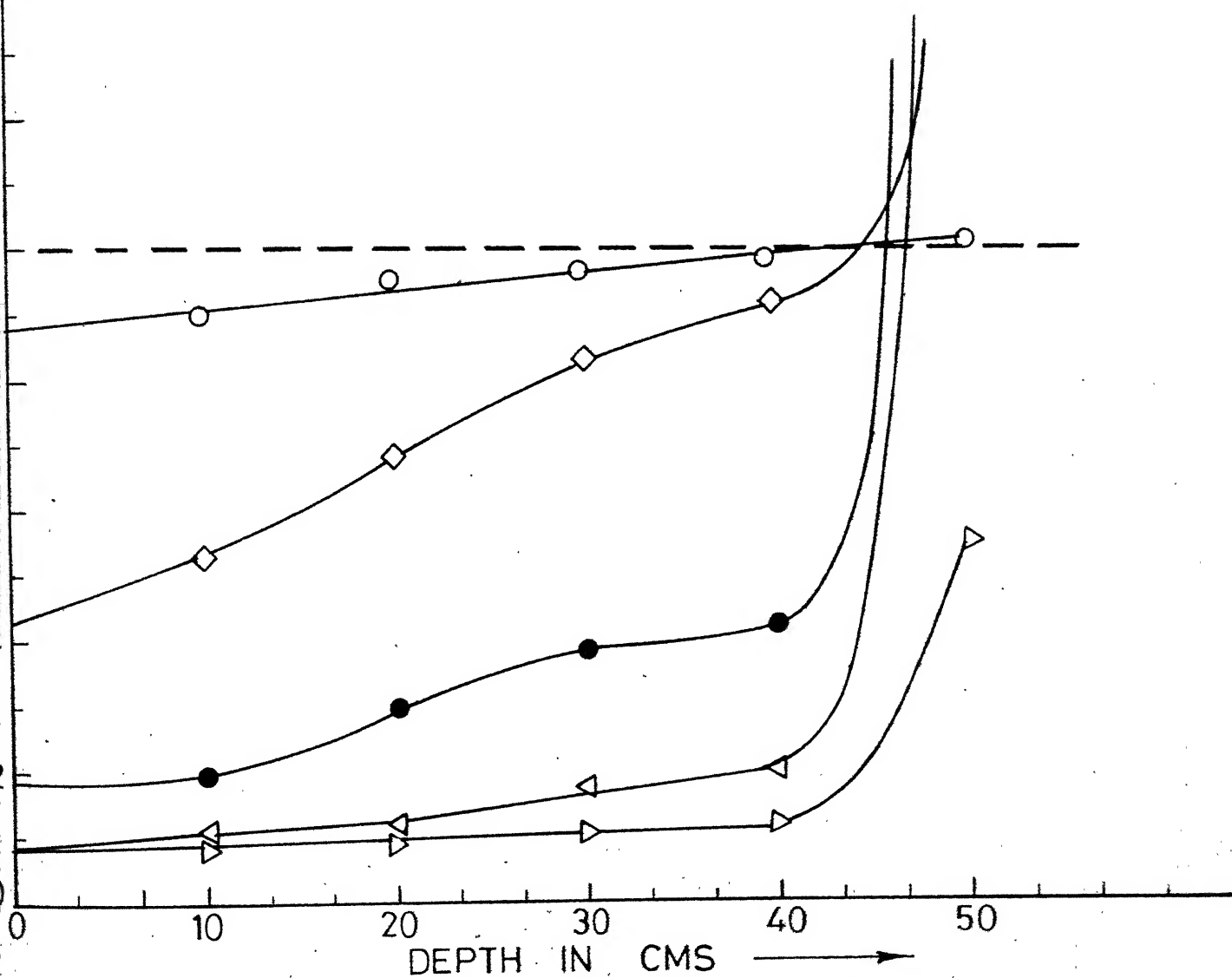
- 0 mt  
 ○ — 1 mt  
 ◇ — 5 mt  
 ● — 15 mt  
 △ — 30 mt  
 ▽ — 60 mt



G. 7. SETTLING CHARACTERISTICS OF KAOLINITE WITH DISPERSANT ( $\text{Na}_4\text{P}_2\text{O}_7$ )

## LEGEND

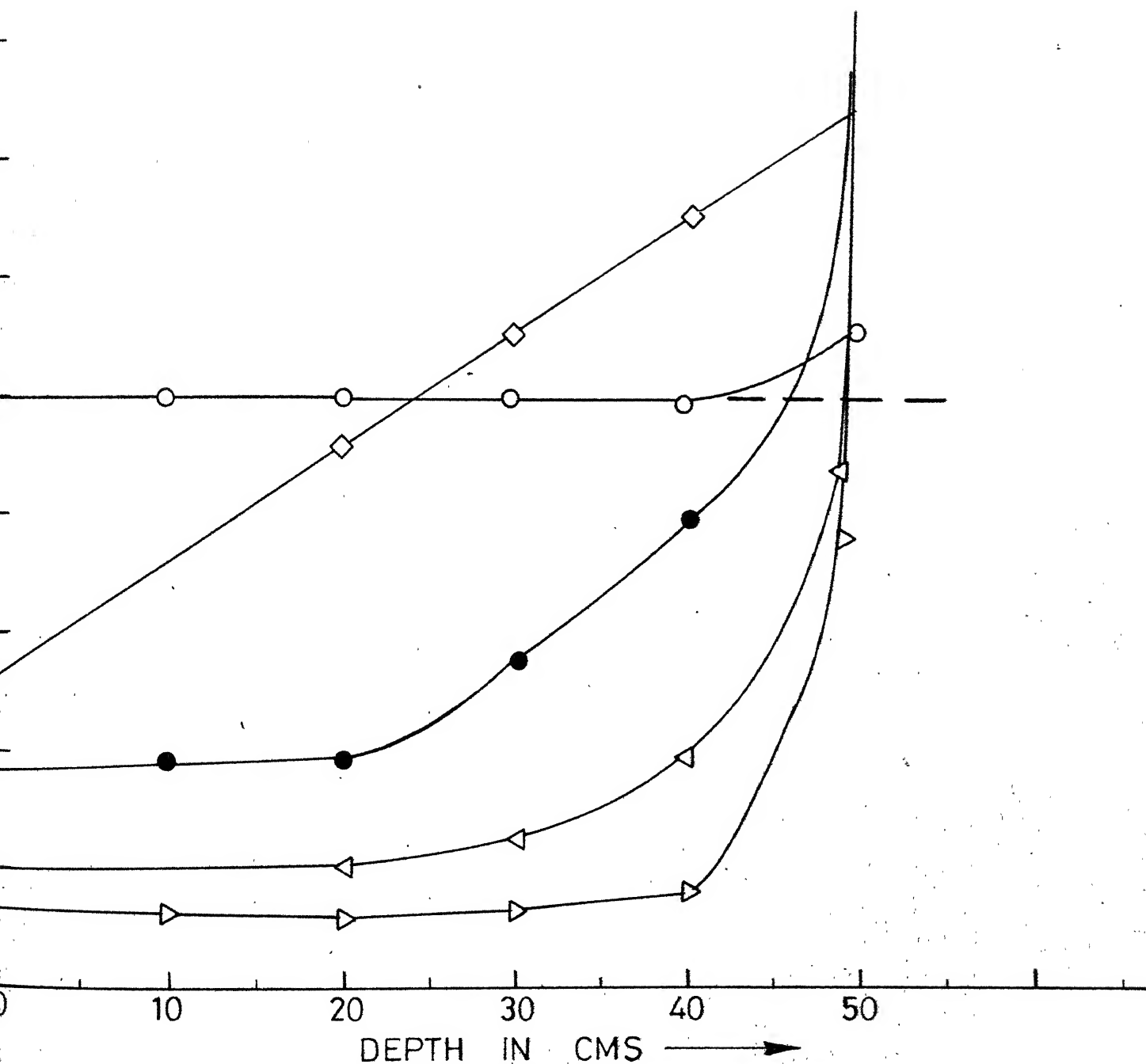
- 0 mt  
 ○ — 1 mt  
 ◇ — 5 mt  
 ● — 15 mt  
 △ — 30 mt  
 ▽ — 60 mt



8 SETTLING CHARACTERISTICS OF KAOLINITE WITH DISPERSANT ( $\text{Na}_2\text{SiO}_3$ )

## LEGEND

- 0 mt  
 ○ 1 mt  
 ◇ 5 mt  
 ● 15 mt  
 △ 30 mt  
 ▽ 60 mt

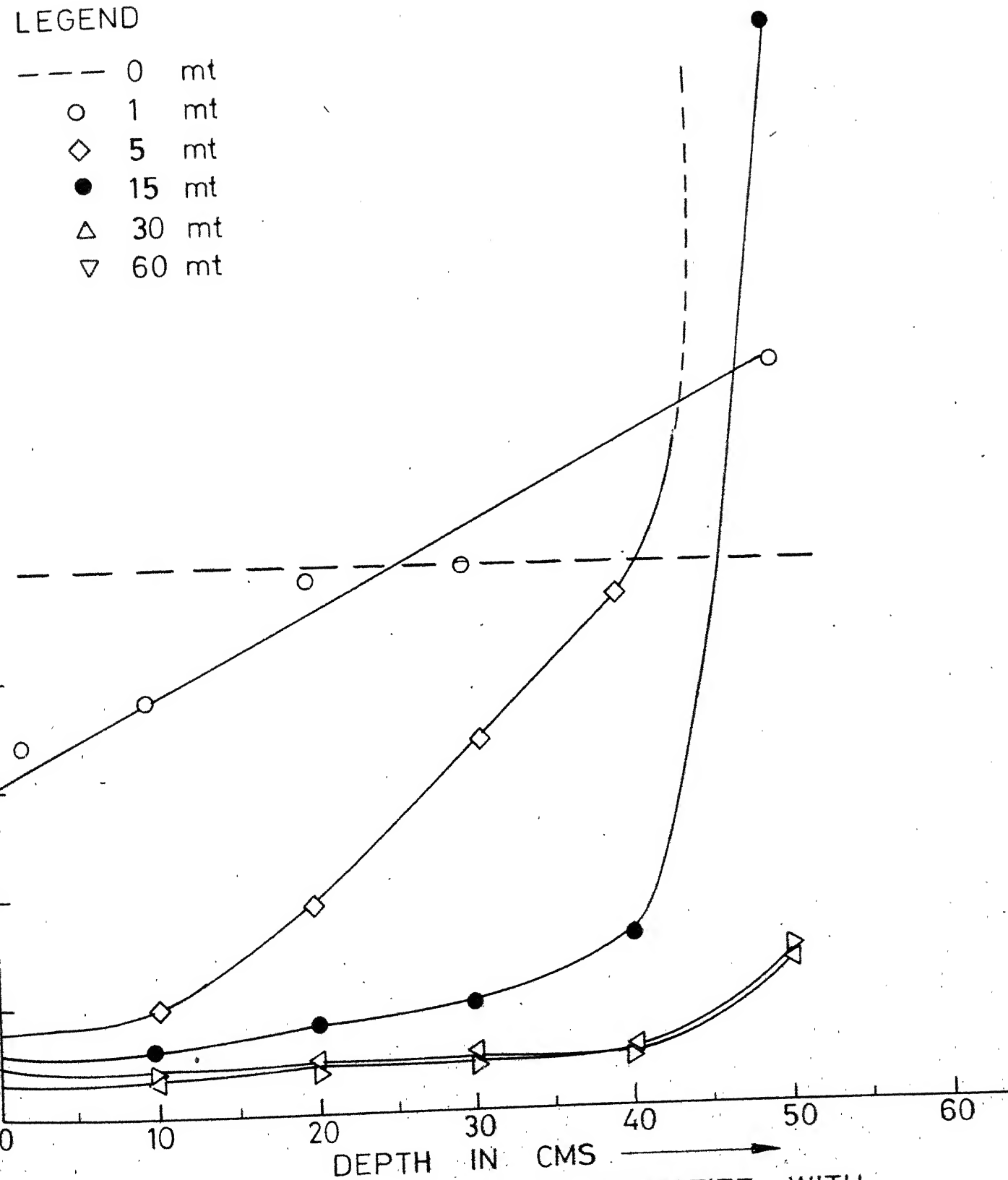


9 SETTLING CHARACTERISTICS OF KAOLINITE WITH DISPERSANT (VERSICOL W 13)



## LEGEND

- 0 mt  
 ○ 1 mt  
 ◇ 5 mt  
 ● 15 mt  
 △ 30 mt  
 ▽ 60 mt



10 SETTLING CHARACTERISTICS OF HEMATITE WITH  
DISPERSANT [(50:50)  $\text{Na}_4\text{P}_2\text{O}_7$  :  $\text{Na}_2\text{SiO}_3$ ]

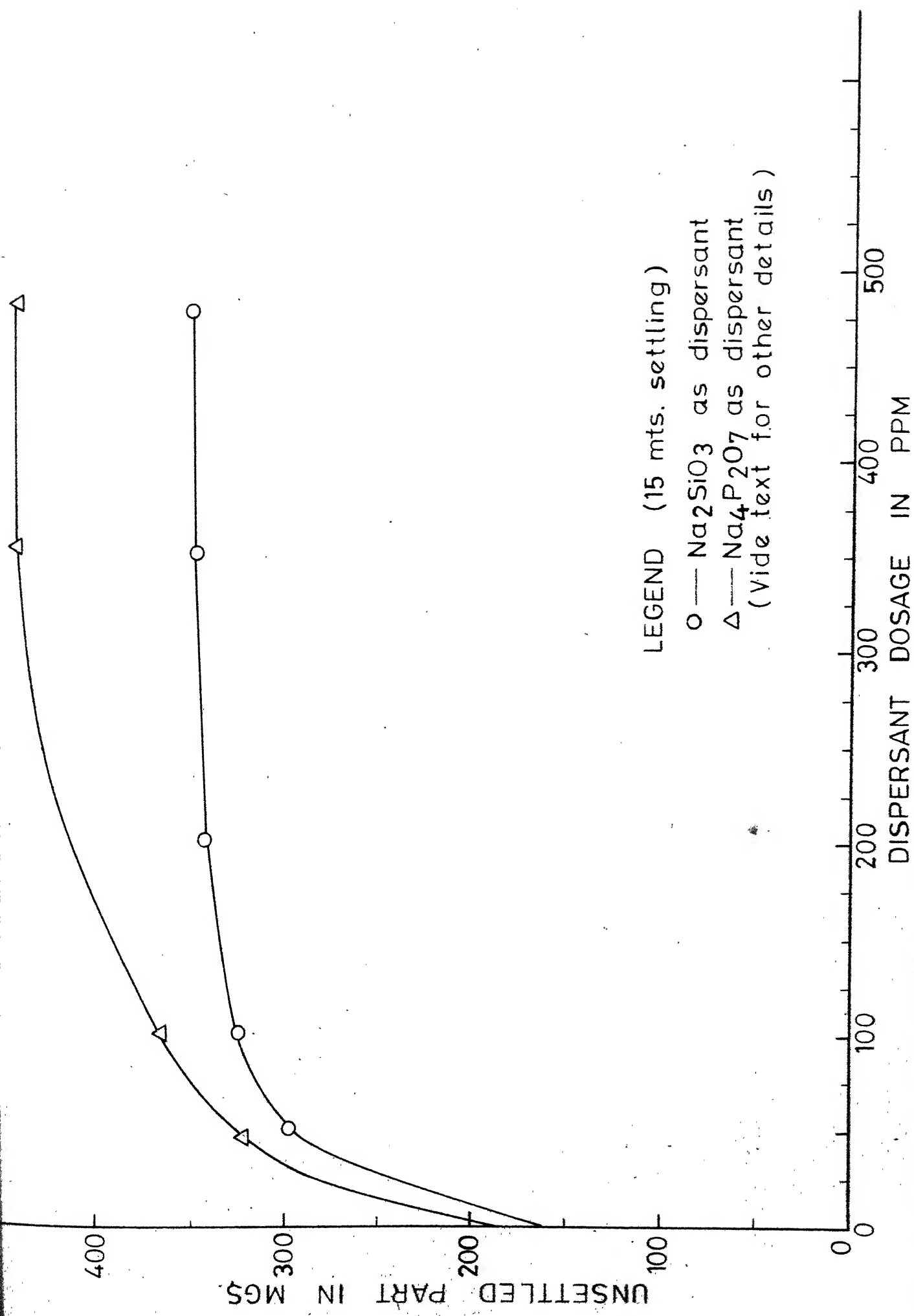


FIG.11 EFFECT OF DISPERSANT DOSAGE ON SETTLING BEHAVIOUR OF MONTMORILLONITE

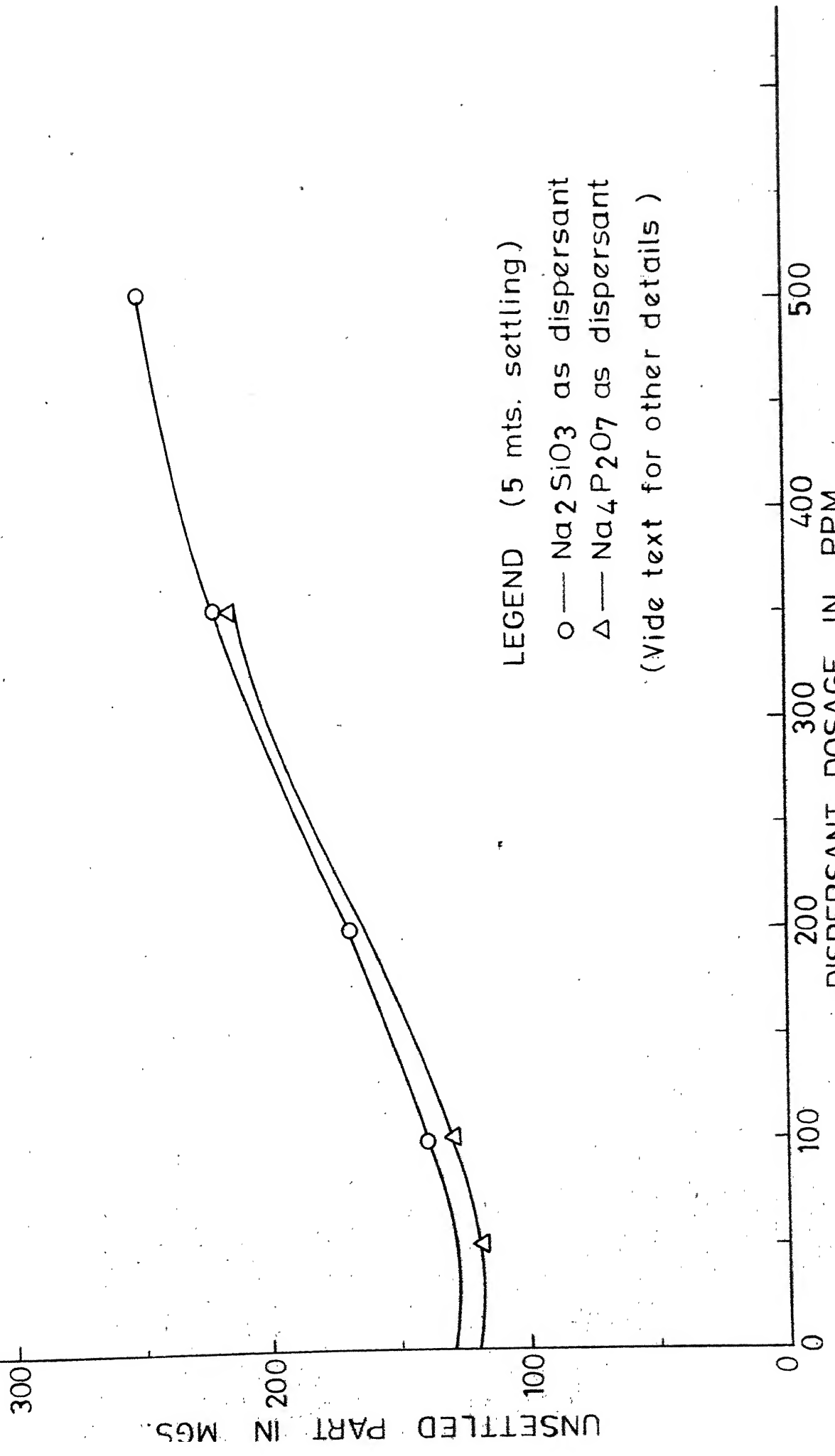


FIG.12 EFFECT OF DISPERSANT DOSAGE ON SETTLING BEHAVIOUR OF HEMATITE.

## CHAPTER VI

## SUMMARY

This report first outlines the results of investigations pertaining to phase-characterisations of the aluminium-containing grains in Barsua (Rourkela) and Bhilai iron ores.

No such investigation seems to have been previously done in India. Previous mineralogical investigations<sup>2</sup> did not result in identification of any aluminium mineral. Even the G.S.I. work on X-ray (Fig. 1) failed to detect any aluminium mineral.

Our painstaking efforts related to specific dissolution of hematite, X-ray and electron microscopy established the presence of (i) Illite, Kaolinite and Montmorillonite (and also possibly Diaspore and Gibbsite) in the Barsua ore and (ii) Sillimanite, Illite, Alunite, Hydrated Aluminium Silicate and Montmorillonite (and also possibly chloritoid, Kyanite, corundum and kaolinite) in the Bhilai ore. Electron probe micro-analysis of the Barsua ore established the elemental composition of some aluminium-containing grains.

The sizes of the aluminium-containing grains varied between 0.03 to 2.0 microns for the Barsua sample and 0.05 to 2.0 microns for the Bhilai sample. Although there could be

bigger grains possibly undetected and some liberation even at coarser sizes due to preferential grinding, it is obvious that very fine grinding is necessary for good liberation. For such slimy particles, selective flocculation is likely to be a promising beneficiation technique.

The oxidized iron formation at the Tilden Mine, owned by Cleveland Cliffs Iron Company, U.S.A., has a grain size of less than 25 microns, and this crude ore is being commercially reduced to approximately 85 minus 500 mesh (25 microns) prior to a beneficiation step involving selective flocculation<sup>15</sup>.

Selective flocculation is one of the most promising avenues for beneficiating fine particles<sup>16</sup> which can not be processed by conventional techniques such as gravity separation, flotation etc. Some basic studies have been made on binary systems of pure minerals and separability of pure minerals by selective flocculation<sup>17,18</sup>. However, it is acknowledged that an ore body is a much more complex system and the results from the studies on binary systems should be translated into plant practice with great caution. In this connection, precise mineralogical characterisation of an ore body becomes a matter of paramount importance. Clayey minerals containing aluminium are easily dispersible with electrolytes having polyvalent anions. It has been seen in the present investigation that appropriate dispersion of the

above minerals by electrolytes and dispersant is a very crucial step. Selective flocculation is possible with cationic polymer (flocculating negatively charged clayey particles) or with anionic polymer (flocculating more of the iron-bearing minerals). Starch is a promising flocculant. Multiple stage flocculation has its obvious merit<sup>16</sup>. The preliminary selective flocculation experiments show distinct promise. Intensive efforts are being made towards achievement of a higher order of selectivity and scientific rationalisation of the results so achieved.

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APPENDIX 'A'

COMMUNICATIONS FROM SOME GLOBAL EXPERTS IN  
MINERALOGY ON OUR PROBLEM

1) Prof. J.A. Kitchener, Royal School of Mines, U.K.

'The precise characterisation of ill-defined iron ores and bauxites is problematical. Although X-ray diffraction indicates the major crystalline components, it says nothing about the amorphous or quasi-crystalline phases .... one must also remember that many clay minerals are capable of incorporating Fe-atoms in the lattice..... As regards a particle size distribution of mixed clay minerals - that is frankly impossible at the present time. For well-crystalline kaolinites, it can be done by long and tedious electron microscopic measurements on a large number of specimens. But illite and montmorillonite particles are so small and poorly discriminated in the electron microscope that quantitative size distributions are not obtainable'.

2) Prof. M.L. Jackson, University of Wisconsin, U.S.A.

'The citrate-bicarbonate-dithionite method<sup>14</sup> (Appendix 'C' in this thesis-author) is very effective in removing hematite from moderately finely ground iron ores. The insoluble then will tell whether the Al is in minerals or other insoluble form. Al can be substituted in iron compounds and would be

dissolved by the CBD treatment. Ordinarily Al goes into the goethite structure more than into hematite'.

- 3) Dr. G.M. Steed and Prof. A.P. Millman, University College, Cardiff, U.K.

'We are not carrying out work of the type you describe. For immediate assistance, you may contact Robertson Research Co. Ltd., Llanddulas, Abergelle, Denbighshire, U.K. We enclose ASTM cards for Diaspore, Boehmite and Gibbsite'.

- 4) Dr. P. Ibbotson, Robertson Research International Ltd.

'We do not have our own electron microscopy equipment and so could not undertake the work on the low cost basis that you require. You may contact Royal School of Mines'.

- 5) Prof. G.W. Brindley, Pennsylvania State Univ., USA

'I have never had to study this particular problem, but one approach would be to remove the iron oxides with the mildest treatment. Kaolinite is unaffected by warm HCl; montmorillonite and illite might be affected so you should use the mildest technique possible'.

- 6) Prof. W.D. Keller, University of Missouri-Columbia, USA.

'Our scan electron microscope equipment does not have high enough resolution and analytical equipment to do the research you need'.

APPENDIX 'B'TYPICAL 'd' VALUES FOR Fe/Al MINERALS<sup>9-13</sup>

ASTM Card No. 13-534

Fe<sub>2</sub>O<sub>3</sub>  
Hematite

d    2.69   1.69   2.51   3.66  
I/I, 100     60     50     25

d Å	I/I,	hkl	d Å	I/I,	hkl
3.66	25	012	1.226	2	036
2.69	100	104	1.213	4	223
2.51	50	110	1.189	8	128
2.285	2	006	1.162	10	0210
2.201	30	113	1.141	12	134
2.070	2	202	1.102	14	226
1.838	40	024	1.076	2	642
1.690	60	116	1.055	18	2110
1.634	4	211	1.042	2	1112
1.596	16	018	1.038	2	404
1.484	35	214	0.9890	10	232, 318
1.452	35	300	.9715	2	229
1.349	4	208			
1.310	20	1010, 119			
1.258	8	220			

ASTM Card No. 17-536

FeO(OH) alpha Form  
Goethite

d    4.18   2.69   2.45   4.98  
I/I, 100     30     25     10

d Å	I/I,	hkl	d Å	I/I,	hkl
4.98	10	020	1.770	2	141
4.18	100	110	1.721	20	221
3.38	10	120	1.694	10	240
2.69	30	130	1.661	4	060
2.58	8	021	1.606	6	231
2.520	4	101	1.564	16	151, 160
2.490	16	040	1.509	10	250, 002
2.452	25	111	1.467	4	320
2.252	10	121	1.453	10	061
2.192	20	140	1.418	2	112
2.009	2	131	1.392	8	330
1.920	6	041	1.357	8	311
1.799	8	211	1.317	8	321

(vi)

1) KAOLINATE (Basic Aluminium  
Silicate) ASTM Card No. 5-0143

$\frac{d}{\text{in } \text{\AA}^\circ}$	$\frac{I}{I_1}$ in%
<u>7.14</u>	100
4.45	50
4.15	60
<u>4.17</u>	<u>60</u>
4.12	30
3.84	40
3.73	20
<u>3.57</u>	100
3.37	40
3.14	20
3.09	20
2.75	20
<u>2.55</u>	70
2.52	40
<u>2.486</u>	80
<u>2.374</u>	70
<u>2.331</u>	90
<u>2.284</u>	80

2) ILLITE ASTM Card No 9-343

$\frac{d}{\text{in } \text{\AA}^\circ}$	$\frac{I}{I_1}$ in%
9.9	80
4.9	60
<u>4.46</u>	100
4.29	40
4.11	40
3.88	60
3.65	50
<u>3.36</u>	100
3.10	50 <sup>B</sup>
2.86	60 <sup>B</sup>
<u>2.57</u>	100 <sup>B</sup>
<u>2.45</u>	50
<u>2.39</u>	<u>60</u>
2.24	50
2.14	60
<u>1.992</u>	60
1.94	40
1.65	60 <sup>B</sup>
1.50	80 <sup>B</sup>
1.345	50 <sup>B</sup>
1.297	60 <sup>B</sup>
1.269	40
1.245	50

3) MONTMORILLONITE: ASTM  
Card No 2-0037

$\frac{d}{\text{in } \text{\AA}^\circ}$	$\frac{I}{I_1}$ in%
<u>11.0</u>	100
5.90	20
<u>4.45</u>	<u>30</u>
<u>2.97</u>	<u>60</u>
2.56	80
2.23	20
1.69	60
<u>1.49</u>	80
<u>1.29</u>	40
1.24	40
1.11	10
1.04	10
0.98	20
0.86	20

4) QUARTZ

$\frac{d}{\text{in } \text{\AA}^\circ}$	$\frac{I}{I_1}$ in%
4.27	50
3.35	100
2.45	40
2.34	40
2.30	35
2.18	35
1.49	30
1.82	55

5) Aluminium Silicate  
Hydrated : 9-421

$\frac{d}{\text{in } \text{\AA}^\circ}$	$\frac{I}{I_1}$ in
10.1	90
4.42	100
3.34	90
2.56	80
2.36	60
2.23	20
2.06	10
1.678	80
1.481	90
1.283	70
1.232	70
1.105	40
1.027	20
0.963	20
0.855	40
0.833	20

6) KYANITE: (Aluminum Silicate)  
ASTM Card No 3-1169

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1}$ in %
4.37	20
3.80	20
3.38	60
3.20	80
3.04	10
2.96	20
2.70	30
2.61	40
2.52	40
2.36	30
2.27	10
2.22 <sup>B</sup>	20
2.16 <sup>B</sup>	20
2.01 <sup>B</sup>	10
1.96	40
1.93	80
1.76	20
1.62	10
1.59	20
1.50	20
1.47	30
1.45	10
1.41	10
1.39	10
1.38	100
1.34	80
1.29	10
1.27 <sup>B</sup>	10
1.26 <sup>B</sup>	10
1.11 <sup>B</sup>	20

7) Calcium Aluminite ( $\text{Ca}(\text{AlO}_2)_2$ )  
ASTM Card No 1-0888

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1}$ in %
4.69	13
2.97	100
2.52	42
2.20	10
1.92	20
1.53	20

8) ALUNITE: 2-0703

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1}$ in %
5.70	20
4.92	40
3.50	20
2.96	100
2.24	50
1.89	50
1.74	50
1.48	60
1.37	20
1.29	20
1.20	20
1.13	20
1.07	10
0.95	10

9) NONTRONITE: (Basic Iron  
Aluminu )  
ASTM Card No 2-0027

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1}$ in %
13.9	100
7.1	20 <sup>B</sup>
4.44	80 <sup>B</sup>
3.54	50
2.79	10
2.59	20-50
2.51	20 <sup>B</sup>
2.17	10 <sup>B</sup>
1.72	20 <sup>BB</sup>
1.68	20
1.63	20-
1.52	50
1.43	50
1.45	10
1.37	10
1.28	10
1.26	20-
1.23	20-

10) Alpha Aluminium Oxide: ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>) 13) GIBBSITE  $\alpha$ -Al(OH)<sub>3</sub>  
CORUNDUM ASTM Card No 10-1733 12-460

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1} \text{ in } \%$
3.479	75
2.379	40
2.085	100
1.740	45
1.601	80
1.404	30
1.374	50
1.239	16
1.0426	14
0.9976	12
0.9178	14

11) SILIMINITE: (Aluminium Silicate) ASTM Card No 1-0614

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1} \text{ in } \%$
5.34	33
3.42	100
3.37	100
2.88	50
2.69	50
2.55	83
2.28	17
2.20	100
2.11	42
1.87	42
1.83	33
1.68	58
1.60	50
1.57	25
1.52	83
1.44	50
1.41	8
1.39	33
1.33	80
1.31	8
1.27	50
1.25	33

12) Chloritoid:  
 ASTM Card No 14-344

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1} \text{ in } \%$
4.47	100
2.46	90
2.97	80

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1} \text{ in } \%$
4.82	100
4.34	40
4.30	20
3.35	10
3.31	06
3.17	08
3.08	04
2.44	16
2.42	04
2.37	20
2.28	04
2.23	06
2.15	08
2.03	12
1.98	10
1.95	02
1.90	08
1.79	10
1.74	10
1.67	10
1.65	04
1.63	02
1.58	04
1.57	02
1.55	02

14) GIBBSITE  $\frac{1}{2}(\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O})$  7-324

$\frac{d}{\text{in } A^\circ}$	$\frac{I}{I_1} \text{ in } \%$
4.35	320
4.37	50
4.32	25
3.506	16
3.187	12
3.112	08
2.454	25
2.420	20
2.388	25
2.285	06
2.244	10
2.168	08
2.085	02
2.043	18
1.993	12
1.960	02
1.921	12
1.799	14

GIBBSITE 7-324 Contd.

$\bar{d}$ in $\text{\AA}^\circ$	I/I <sub>1</sub> in %
1.750	16
1.689	14
1.654	04
1.638	02
1.593	04
1.584	02
1.573	04
1.555	02
1.551	02
1.486	02
1.477	02
1.457	10
1.441	06
1.409	06
1.398	06
1.380	02
1.361	04
1.340	02
1.330	02
1.320	04
1.246	02
1.214	04

DIASPORE 5-355

AlO(OH)

$\bar{d}$ in $\text{\AA}^\circ$	I/I <sub>1</sub> in %
4.71	13
3.99	100
3.214	10
2.558	30
2.434	03
2.386	05
2.356	08
2.317	56
2.131	52
2.077	49
1.901	03
1.815	08
1.733	03
1.712	15
1.678	03
1.633	43
1.608	12
1.570	04

DIASPORE Contd.

$\bar{d}$ in $\text{\AA}^\circ$	I/I <sub>1</sub> in %
1.522	06
1.480	20
1.431	07
1.423	12
1.400	06
1.376	16
1.340	05
1.329	06
1.304	03
1.289	06
1.229	01
1.256	04
1.243	05
1.218	02
1.204	04
1.1739	07
1.1783	01
1.1408	03
1.1003	01
1.0923	03

BOEHMITE 21-1307 $\gamma$ -AlOOH

$\bar{d}$ in $\text{\AA}^\circ$	I/I <sub>1</sub> in %
6.11	100
3.164	65
2.346	65
1.980	06
1.860	30
1.850	25
1.770	06
1.662	14
1.527	06
1.453	16
1.412	10
1.396	02
1.383	02
1.369	06
1.312	16
1.303	04
1.224	02
1.209	02
1.178	04
1.1711	02

(x)

BOEHMITE

Contd.

$\bar{d}$ in $\text{\AA}^\circ$	$I/I_1$ in %
1.1609	04
1.1337	06
1.1152	02
1.0917	02
1.980	06
1.860	30
1.850	25
1.770	06
1.662	14
1.0459	02
1.0281	02
0.9903	02
0.9818	02
0.9506	02
0.9310	02
0.9247	02
0.9105	02
0.9023	02
0.8937	02
0.8907	02
0.8660	02
0.8607	02
0.8316	02
0.8286	04



APPENDIX 'C'REMOVAL OF IRON OXIDE FROM IRON ORE (HEMATITE)AS SUGGESTED BY M.L. JACKSON<sup>14</sup>

PROCEDURE: A known weight (1 gm) of the iron ore was taken in a cylindrical vessel and to it was added 200 c.c. of 0.3M Na citrate together with 1M  $\text{NaHCO}_3$  solution. The container fitted with a glass stirrer inside was placed in a water bath whose temperature was raised previously to 65-70°C. Finally the temperature was raised to 75-80°C with the stirrer rotating at maximum possible speed. 5 gm of  $\text{Na}_2\text{S}_2\text{O}_4$  was added and vigorous, stirring was continued. After about 5 minutes another 5 gm of  $\text{Na}_2\text{S}_2\text{O}_4$  was added and stirring continued 25 c.c. of acetone and 25 c.c. of saturated NaCl were added after five minutes. The suspension was allowed to settle for 30-45 seconds and the supernatant liquid was decanted. Solid black particles were transferred to a beaker. This procedure was repeated for further quantities of the sample.

The collected particles were finally dried by infrared heating.

Approximately 12.6% of the mass taken was collected as blackish particles supposed to <sup>be</sup> hematite free.

## APPENDIX 'D'

MEASURED 'd' VALUES FOR STANDARD AND UNKNOWN SPECIMENS

FIGURE 3

HEMATITE (H)	KAOLINITE (K)	ILLITE (I)	MONTMORILL ONITE (M)	RESIDUE AFTER LEACHING <sup>14</sup>
1.308	1.492	1.229	1.372	1.314 * (H)
1.452	1.542	1.372	<u>1.497</u>	1.456 * (H)
1.483	1.621	1.541	<u>1.512</u>	1.487 * (H)
1.600	1.665	1.602	<u>1.668</u>	1.601 * <sub>1</sub>
1.696	1.790	1.674	1.821	1.697 * (H)
1.843	1.846	1.820	2.458	1.720 * <sub>2</sub>
2.209	1.943	1.894	<u>2.565</u>	1.738
2.520	1.991	<u>1.980</u>	<u>2.577</u>	1.754
2.701	<u>2.304</u>	<u>2.129</u>	3.041	1.801
3.676	<u>2.348</u>	<u>2.379</u>	3.114	1.843 * (H/Q)
	<u>2.389</u>	<u>2.452</u>	3.151	1.922
	<u>2.508</u>	<u>2.579</u>	3.247	1.991
	<u>2.543</u>	<u>2.685</u>	3.353	2.052
	<u>2.573</u>	<u>2.969</u>	3.524	2.211 * (H)
	3.380	3.132	3.792	2.253
	<u>3.598</u>	<u>3.349</u>	3.971	2.389 * (K/I)
	<u>3.867</u>	<u>3.548</u>	4.276	2.455 * (I/M)
	<u>4.199</u>	4.005	4.374	2.526 * (H)
	<u>4.394</u>	4.272	<u>4.4891</u>	2.591 (I)
	<u>7.241</u>	4.888	<u>4.690</u>	2.709 * (H)
		5.451	4.776	3.334 * (I/M/Q)
		7.241	5.054	3.673 * (H)
		7.671		4.167 * <sub>3</sub> (K)
		8.706		4.358 * <sub>3</sub>
				4.820 * (G)
				7.241 (K)

Q...Quartz      G....Goethite

\* ... Sharp peaks

- 1 H/I/Corundum/Sillimanite or Diaspore
- 2 G/or Nontronite
- 3 Kyanite or Gibbsite

APPENDIX 'E''d' values from electron diffraction patterns taken  
from BARSUA SAMPLES

$\lambda = 0.037$   $L = 120$  cms  $L\lambda = 4.44$  'R' is radius of the  
ring

A) Diffraction Pattern No. 603. Corresponding electron  
micrograph no. 604

'R' values in cms	$\frac{L\lambda}{R} = d$ in $\text{\AA}^\circ$	Likely mineral with d values
1.925	2.307	Quartz (2.30)
1.800	2.467	Quartz (2.45)
2.800	1.586	Kaolinite (1.59)
1.600	2.775	" (2.75)
2.350	1.889	-
2.750	1.615	Kyanite (1.62)
2.40	1.85	Hematite (1.84)
2.65	1.675	Gibbsite (1.67) Diaspore (1.68)
3.35	1.325	Illite (1.323) Gibbsite (1.320)
2.15	2.041	Al-silicate hydrated (2.06)
1.85	2.40	Illite (2.42)
2.70	1.644	Illite (1.65)
2.45	1.812	Quartz (1.82)
1.95	2.277	Kaolinite, Diaspore, Gibbsite (2.28)
3.55	1.2507	Illite (1.245)

B) Diffraction Pattern No. 605. Micrograph 606

1.925	2.307	Quartz (2.30)
1.825	2.433	Quartz (2.45), Diaspore (2.434)
2.00	2.22	Montmorillonite (2.23)
2.85	1.5579	Gibbsite (1.555)
3.25	1.366	Quartz (1.372)
3.125	1.421	Diaspore (1.423)
3.15	1.41	Kyanite (1.41)
3.40	1.306	Diaspore (1.304)
4.05	1.096	-
3.725	1.192	Quartz (1.191)

C) Diffraction Pattern 607. Micrograph 608

1.825	2.433	Diaspore/Quartz (2.434)
1.95	2.277	Gibbsite/Kaolinite (2.28)
3.175	1.390	
3.15	1.4095	Kyanite (1.41)
3.25	1.366	-
3.35	1.325	Illite (1.323)

D) Diffraction Pattern 609. Micrograph 610

2.00	2.22	Montmorillonite (2.23)
2.175	2.041	-
2.325	1.91	Diaspore (1.901)
3.275	1.3557	Illite (1.345)

E) Diffraction Pattern 611. Micrograph 612

2.675	1.656	Illite (1.65)
2.55	1.7412	Gibbsite (1.74)
2.500	1.776	-

American cyanamide manufactures Aerofloc 550<sup>\*</sup> - a dry solid useful in all pH except acidic (as 20% solution - and also Superfloc 202 (very high molecular weight polyacrylamide useful in coal flotation) and also Aerofloc 3425 and 3453. Dow chem manufactures Separan AP 30. B.T.I. chemicals, Bradford supplies polyacrylamides of different anionic charge density (0, 10 ... 80 p.c. amide-carboxylate substitution) - by hydrolysing amide groups to different extent, degree of polymerisation being  $2-4 \times 10^4$ . Flocculants have typical concentrations as 0.01 to 0.1% which is equivalent to about 0.01 lb/ton of dry solid.

The Dharamsi Morarji chemical Co. Ltd., Jolly Bhavan No. 2, New Marine Lines, Bombay-400020 manufactures polyacrylamide type flocculants such a Morarfloc PG, A-30, A-40H and A 100 - the last one being<sup>#</sup>corrosive. These are claimed to be useful in settling of red mud (along with starch), settling of gypsum in phosphoric acid manufacture, clarification of brine, iron ore slimes etc. In a leading iron and steel industry in India, 0.75 ppm of Morarfloc reduces alum requirement from 25 to 10 ppm regarding clarification of solids.

Anil Starch Products, Ahmedabad provides oxidised starch Anilox AP, Anilox AT as well as Maize Starch. Commercial starch supplied by Maize Products Ltd, India, assayed 0.012 pct P. Chemically pure starch containing 0.014 pct P has been phosphorylated by five different techniques to obtain products varying in 0.077 to 4.9 p.c. phosphorus content.

Cationic Flocculants manufactured by Allied Colloids limited are Magnafloc 140<sup>\*</sup>, 292<sup>\*</sup>, 352, 455, HR 120<sup>\*</sup> (liquid) etc. These are supposed to be very suitable for negative charged slimes like clay and silicates. American Cyanamide product Superfloc 310 is a high-molecular weight flocculant useful in low dosage. Nalco 600 - an ethylene diamine product is another popular cationic flocculant. Anil Starch Products Co. manufacture cationic starch which contains tertiary amino groups introduced through reaction with 2-diethyl aminoethyl chloride in alkaline condition.

Nonionic Flocculants serve as multipurpose flocculants in diverse pH conditions e.g., Magnafloc 351<sup>\*</sup>. Another all pH flocculant Superfloc 16<sup>\*</sup> is marketed by American Cyanamide Co. This is a dust-free solid, Superfloc 20, 84, 127 have increasing molecular weights. Separan NP 10<sup>\*</sup> is a popular nonionic flocculant marketed by Dow Chem., Separan NP 20 being a higher molecular weight product. Morarfloc A, TT are indigenous nonionic flocculants, marketed by Dharamsi Morarji Chemical Co. Ltd.